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ANNUAL REPORT

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

for

CHARACTERIZATION OF THE PHYSICO-CHEMICAL PROPERTIES

OF POLYMERIC MATERIALS FOR AEROSPACE FLIGHT

(NASA-CR-156116) CHARACTERIZATION OF THE PHYSICO-CHEMICAL PROPERTIES OF POLYMERIC MATERIALS FOR AEROSPACE FLIGHT Annual Report (Bowie State Coll., Md.) 85 p HC A05/MF A01 CSCL 11G G3/27 N78-20334 Unclas 11169

Michael Rock

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March 17, 1978

Date

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Bowie State College
Bowie, Maryland 20715

INTRODUCTION

The major source of contamination in spacecraft is internal. Outgassing of polymers, greases, and other organic materials at low pressures in space and test causes contamination. Condensation of contaminants on cold surfaces, such as particle detectors or optical surfaces will seriously reduce the efficiency of such units and give erroneous results. In addition, excessive outgassing may lead to corona inception and multipacting and can modify physical properties of the polymers.

Materials intended for use in spacecraft are routinely tested at low pressures, and outgassed substances are condensed on cold surfaces at liquid nitrogen temperature - 196° C. These condensates are then analyzed by infra-red spectroscopy, and gas chromotography-mass spectroscopy.

At GSFC, the polymers are tested and allowed to outgas isothermally at 125° C and 10^{-6} torr or less. Valuable information can be obtained by extending the temperature range below and above 125° C. The performance, of substances of interest to NASA, was investigated by thermogravimetric analysis (TGA) from room temperature to 450° C or until the substance decomposed.

Thermogravimetric analysis of a substance is a useful technique for studying its thermal nature in both static and dynamic thermal environments. It gives considerable insight into the stability and characterization of substances and the changes they undergo in varying thermal environments.

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PROCEDURE

Thermogravimetric analyses are run on a DuPont 950 Thermogravimetric Analyzer. With the final completion of the moving of the equipment and shifting to a vacuum atmosphere, all runs were performed at standard pressure of about .03 microns (3×10^{-5} torr).

A new procedure is currently in use with sample #6995. The new thermal program proceeds as follows:

-- Each sample is run isothermally at ambient temperature (20° C to 30° C) for one-half hour or ten minutes after reaching minimum pressure.

-- The temperature is then programmed to rise at maximum rate (approximately 50° C/min.) to the isotherm temperature. The recorder is set to measure temperature vs. weight during this phase. We thus measure overshoot as the sample is heated. We routinely mark the graph at 60 second intervals during the heat-up phase. This is accomplished by momentarily depressing the weight axis null button.

-- The sample is then held isothermally at the desired temperature (125° C, 200° C, 250° C) for 24 hours and the weight is measured as a function of time. The final step, the temperature decomposition phase, is programmed to rise at 3° C to 4° C per minute from the established isothermal temperature to 450° C or until the sample decomposes.

-- The four thermal steps are:

1. Room temperature isotherm.
2. Room temperature to isotherm temperature, with the unit previously set to stop at the desired isotherm temperature.
3. Isothermally at desired temperature for 24 hours.

4. Sample is heated from isotherm (125° C, 200° C, 250° C) to decomposition.

DISCUSSION OF TABLES

The following is an explanation of colums 3, 4, 5, 6, 7, and 8 in the tables:

Column 3	Shows the weight loss of the sample at ambient temperature - (20° C - 30° C)
Columns 4 and 5	As the sample is heated to the isotherm temperature, the rate of weight loss increases. This rapid rate of weight loss persists for a short time after the isotherm temperature has been reached. The rate of weight loss then decreases and levels off. There appears to be a loss of volatiles during the heating phase with some of the volatiles coming off after the isotherm has been reached. These two portions, that which comes off before the isotherm is reached, and that which comes off at the isotherm, are listed separately in columns 4 and 5. The sum of columns 4 and 5 gives the total weight of these volatiles lost.
Column 6	After the weight curve levels off, there is a continuing slow loss of weight at the isotherm. This weight loss is measured for one day, and the values are listed in column 6.

Columns 7 and 8

After one day, the temperature is programmed to rise at 3° C to 4° C per minute. The recorder graphs temperature vs. weight. The graph starts as a straight line; at some temperature the graph curves down, meaning that the rate of weight loss is increasing. The straight line portion of the graph is extrapolated. Column 7 lists the temperature at which the line curves down; the temperature at which the rate of loss is noticeably greater than the constant rate. Column 8 lists the temperature at which the actual curve is 0.2mg (or 0.2 inches) below the extrapolated line. This measurement is an arbitrary one and was chosen because it is less subjective than the figure in column 7 and gives another point of comparison between different samples.

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FOLDOUT FRAME 1

SAMPLE AND #	WEIGHT	LOSS C ROOM TEMP	LOSS R.T TO 125° C	LOSS TO LEVEL OFF
69 (RTV-600	102	.04 mg	.08 mg	0.13 mg
70 (Solithane (100° C)	60.3 mg	0.11 mg	.07 mg	0.59 mg
71 (RTV-600	97.7 mg	0.1 mg	0.1 mg	.91 mg
72 (Untreated Ne 102	75.3 mg	0.1 mg	---	---
73 (Untreated Ne 102	73.1 mg	.05 mg	---	.05 mg
74 (Ne 102)	65.1 mg	00	---	---
75 Ne Untreated 102	99.4 mg	.38 mg	---	---
76 Ne 102 Baked	103.7 mg	---	---	.08 mg
79 Ne Untreated 102	98.02 mg	---	---	.16 mg
81 NE 102 Baked	98.02 mg	---	---	.16
82 Ne 110	101.28 mg	---	---	see # 83
83 Ne 110	97.56 mg	---	---	.2 mg
84 1018 tape	101.68 mg	---	---	.22 mg
88 1018 tape	88.25 mg	---	---	---
89 Untreated 0080 tape	99.35 mg	---	---	---

FOLDER PLATE 2

LOSS R.T TO 125° C	LOSS TO LEVEL OFF	LOSS @ 125° C	TEMP. DECAY	TEMP. 0.2 mg DEPARTURE
.08 mg	0.13 mg	0.26 mg	170° C	708° C
.07 mg	0.59 mg	0.20 mg	358° C	365° C
0.1 mg	.91 mg	1.0 mg	----	----
---	---	0.1 @ 45° C	100° C	110°C
---	.05 mg	---	105° C	115° C
---	---	---	---	---
---	---	.1 @ 45° C	---	---
---	.08 mg	.06 mg @ 45° C	110° C	125° C
---	.16 mg	.02 mg	105° C	133° C
---	.16	.02 mg @ 45° C	180° C	225° C
---	see # 83	see # 83	210° C	268° C
---	.2 mg	.04 @ 40° C	137° C	155° C
---	.22 mg	.04 mg	---	---
---	---	---	100° C	130° C
---	---	---	115° C	135° C

SAMPLE & #	WEIGHT	LOSS @ ROOM TEMP. .0	LOSS R.T TO 125° C	LOSS TO LEVEL OFF
# 90 Untreated 0080 Tape	99.2 mg	---	---	---
# 91 Untreated 1018 Tape	80.7 mg	---	---	---
#92 Untreated 0080 Tape	78.1 mg	---	---	---
# 94 Untreated 0080 Tape	89.5 mg	---	---	---
# 95 1018 Tape	91.54 mg	---	---	---
# 96 1018 Tape	121.25 mg			
#97 1018 Tape	112.5 mg	---	---	---
# 98 1018 Tape	112.5 mg	---	---	---
# 99 0080 Tape	113.0 mg	---	---	---
#101 0080 Tape	93.5 mg	---	---	---
#102 0080 Tape	64.8 mg	---	---	---
#106 Black Graphite Cloth	69.7 mg	--	to 105° .4 mg	---
#107 Adhesive (Epoxy)	40.6	.3 mg	---	---
#107a Black Graphite Cloth	43.90	.1 mg	to 105° .0 mg	.6 mg

FOLDOUT FRAME 1

LOSS R.T TO 125° C	LOSS TO LEVEL OFF	LOSS @ 125° C	TEMP. DECAY	TEMP. 0.2 mg DEPARTURE
---	---	---	90° C	125° C
---	---	---	100° C	150° C
---	---	---	90° C	130° C
---	---	---	105° C	127° C
---	---	---	100° C	125° C
			120° C	125° C
---	---	---	265°	310°
---	---	---	see # 97	see # 97
---	---	---	100° C	130° C
---	---	---	100° C	130° C
---	---	---	see #94	see #94
to 105° .4 mg	---	.2 mg	---	---
---	---	---	---	---
to 105° .0 mg	.6 mg	105° .08 mg	150° C	350° C

FOLDER FRAME

2

# SAMPLE AND #	WEIGHT	LOSS @ ROOM TEMP.	LOSS R.T TO 125° C	LOSS TO LEVEL OFF
# 108 Adhesive (Epoxy)	42.9 mg	.32	---	---
# 109 Epoxy Adhesive	109.85 mg	.70 mg	@ 105° C 0 mg	.70 mg
# 110 RTV 560/9811	160.9 mg	.05 mg	---	---
#111 RTV-560/9811	115.25 mg	.8 mg	.1 mg	---
#112 RTV-560-9811	116 mg	.9 mg	---	---

POOR QUALITY FRAME

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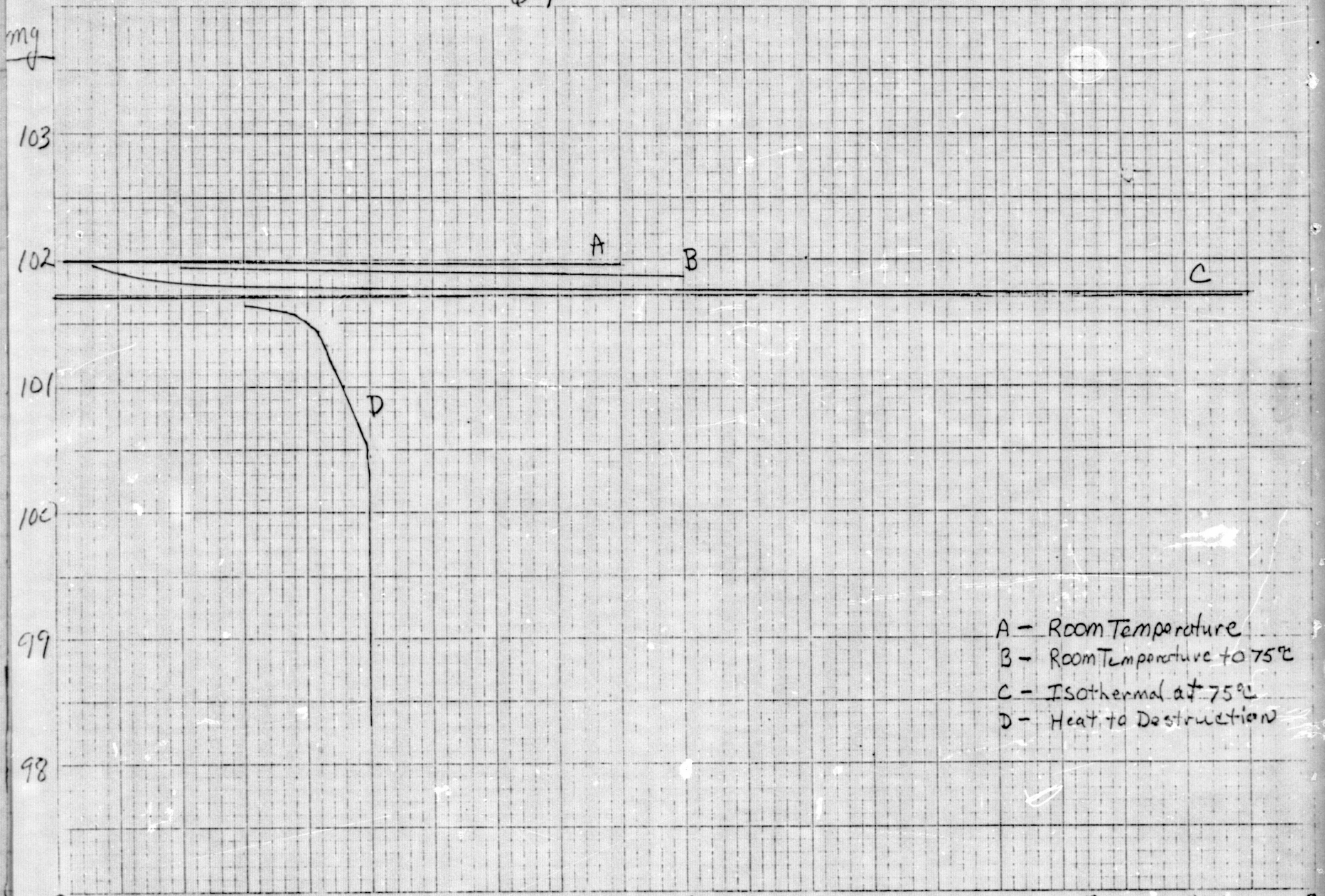
LOSS R.T TO 125° C	LOSS TO LEVEL OFF	LOSS @ 125° C	TEMP. DECAY	TEMP. 0.2 mg DEPARTURE
---	---	---	---	---
@ 105° C O mg	.70 mg	@ 105° C 0	200° C	215° C
---	---	---	---	---
.1 mg	---	---	---	---
---	---	---	---	---

FOLDOUT FRAME

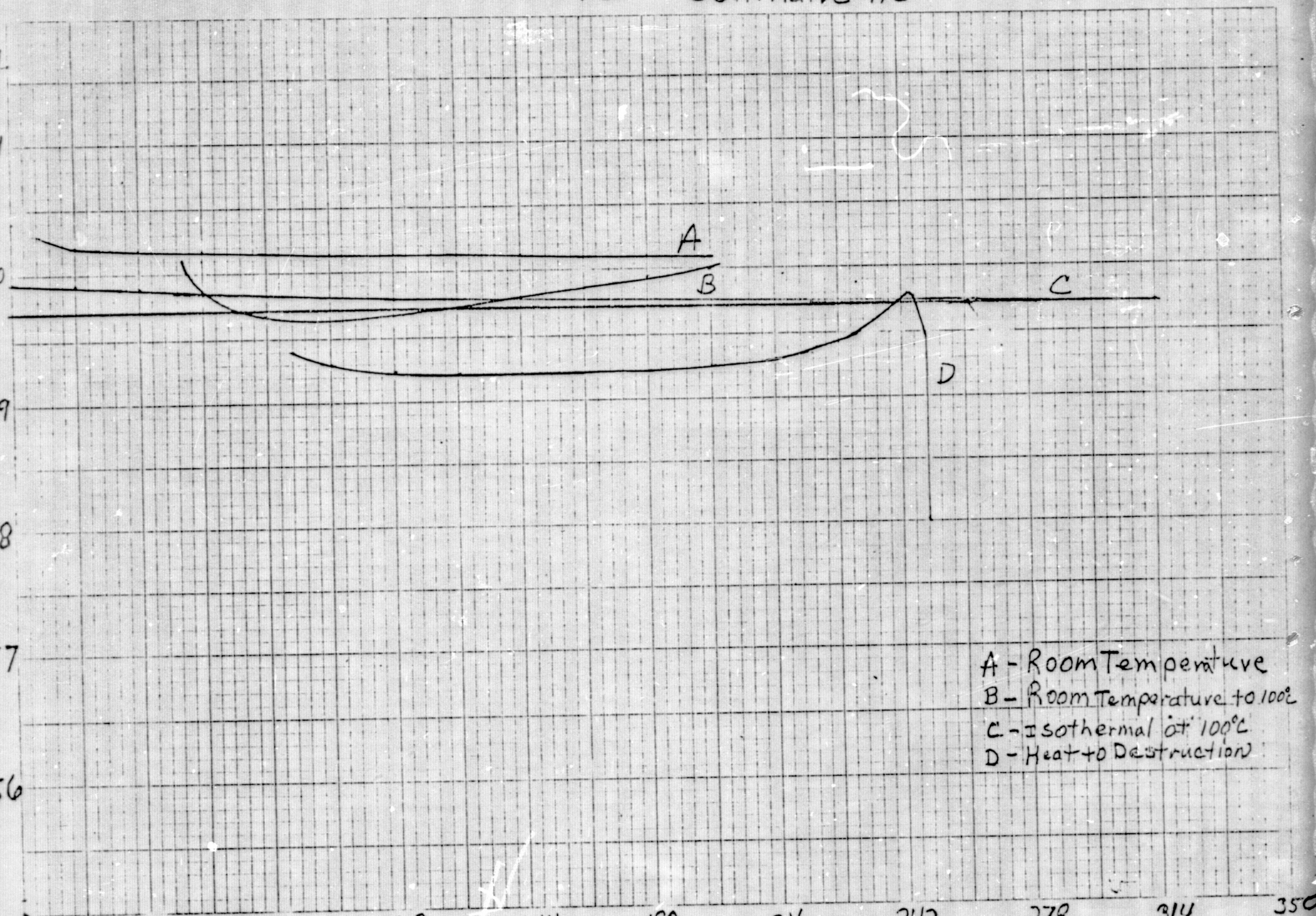
2

#69 RTV-600



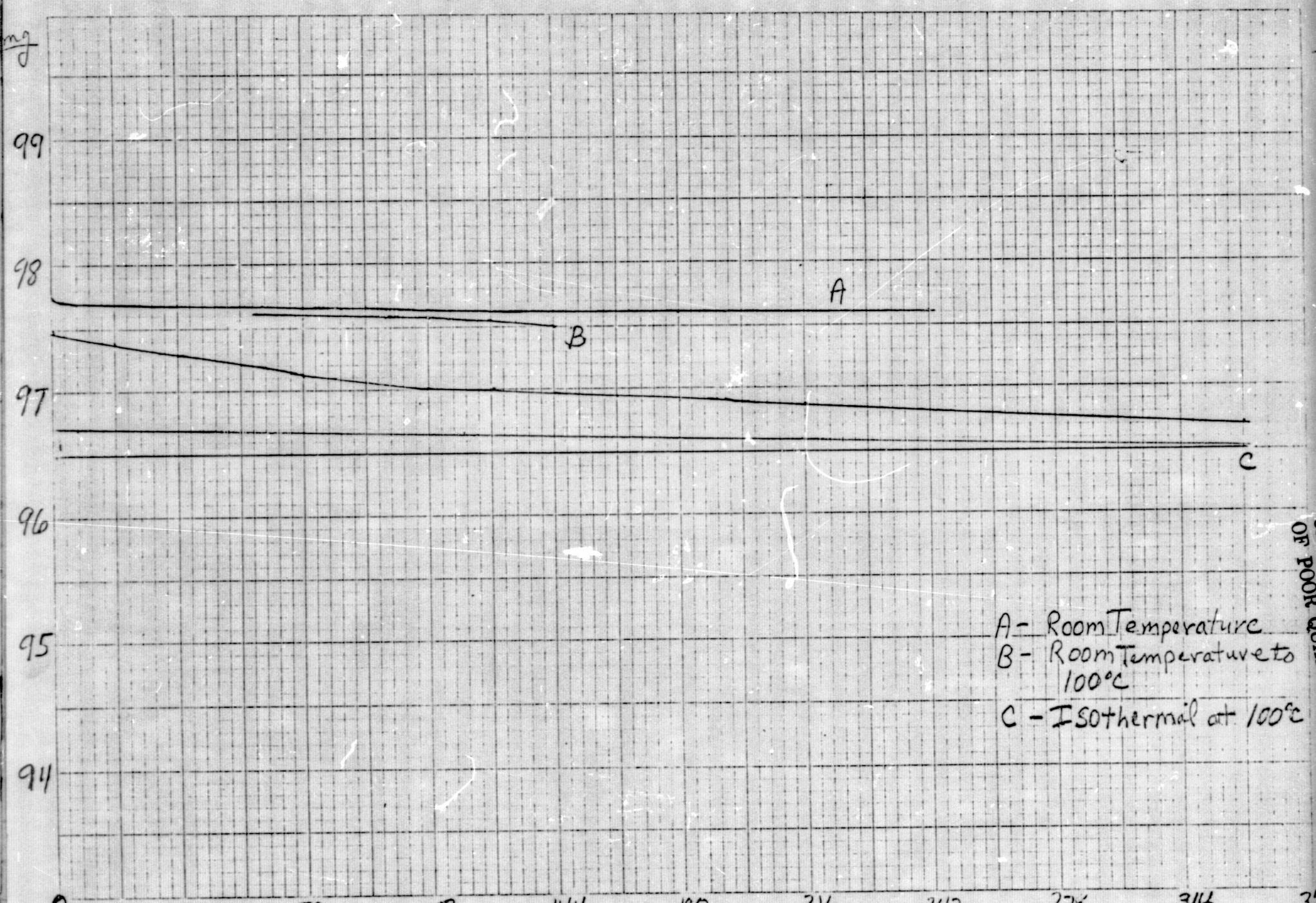
0										
A (mins Per Cycle)	36	72	108	144	180	216	242	278	314	35
B °C	50	100	150	200	250	300	350	400	450	50
C (mins PC)	36	72	108	144	180	216	242	278	314	35
170°C.										

#70 Solithane 113



0	36	72	108	144	180	216	242	278	314	350
A (mins per cycle)	20	40	60	80	100	120	140	160	180	200
B ½ °C					180				3¼	350
C ¼ mins	36	72	108	144		216	242	278		500
			150	200		300	350	400	450	

#11 KIV-600



A - Mins per cycle	0	72	108	144	180	216	242	278	314	350
B - °C	50	100	150	200	250	300	350	400	450	500
C - mPC	36	72	108	144	180	216	242	278	314	350
D - °C	-	-	-							

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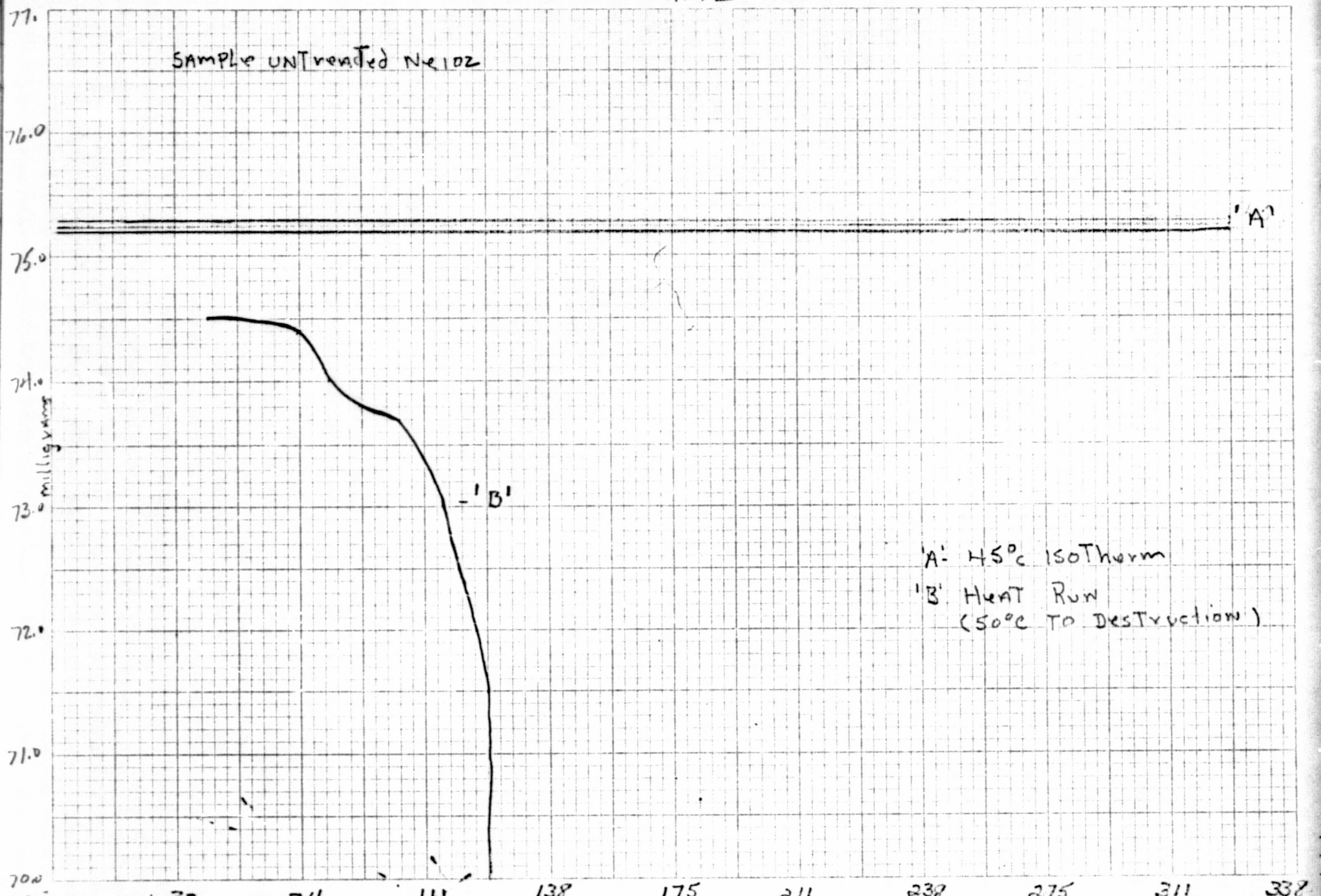
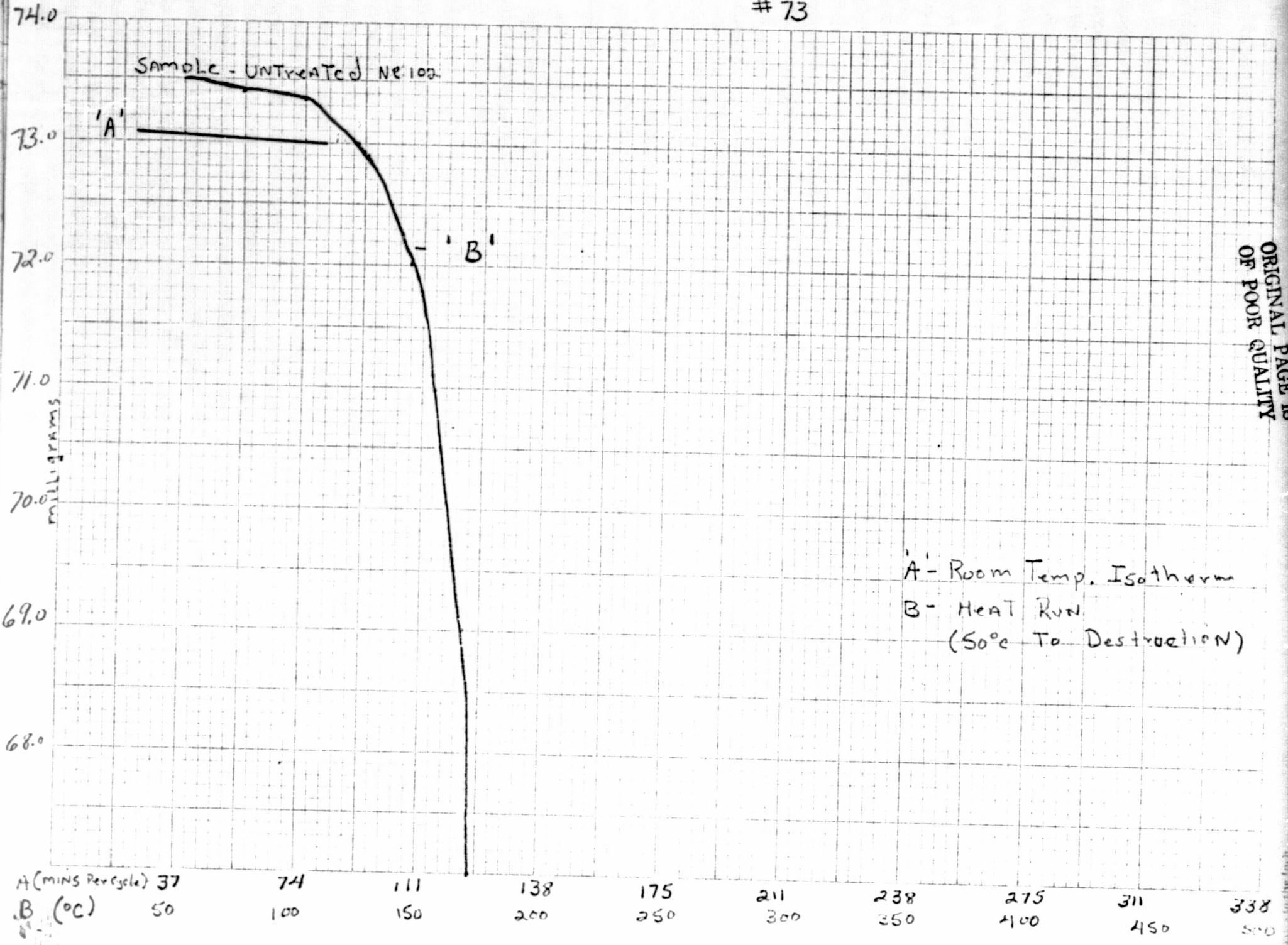
#72


SAMPLE UNTreated Ne102
77.
76.0
75.0
74.0
73.0
72.0
71.0
70.0
milligrams
'A'
'B'
'A' 45°C ISOTherm
'B' HeaT Run
(50°C TO DesTruction)
A (mins per cycle 37 74 111 138 175 211 238 275 311 338
100 150 200 250 300 350 400 450 500
B (°C) 50

#73



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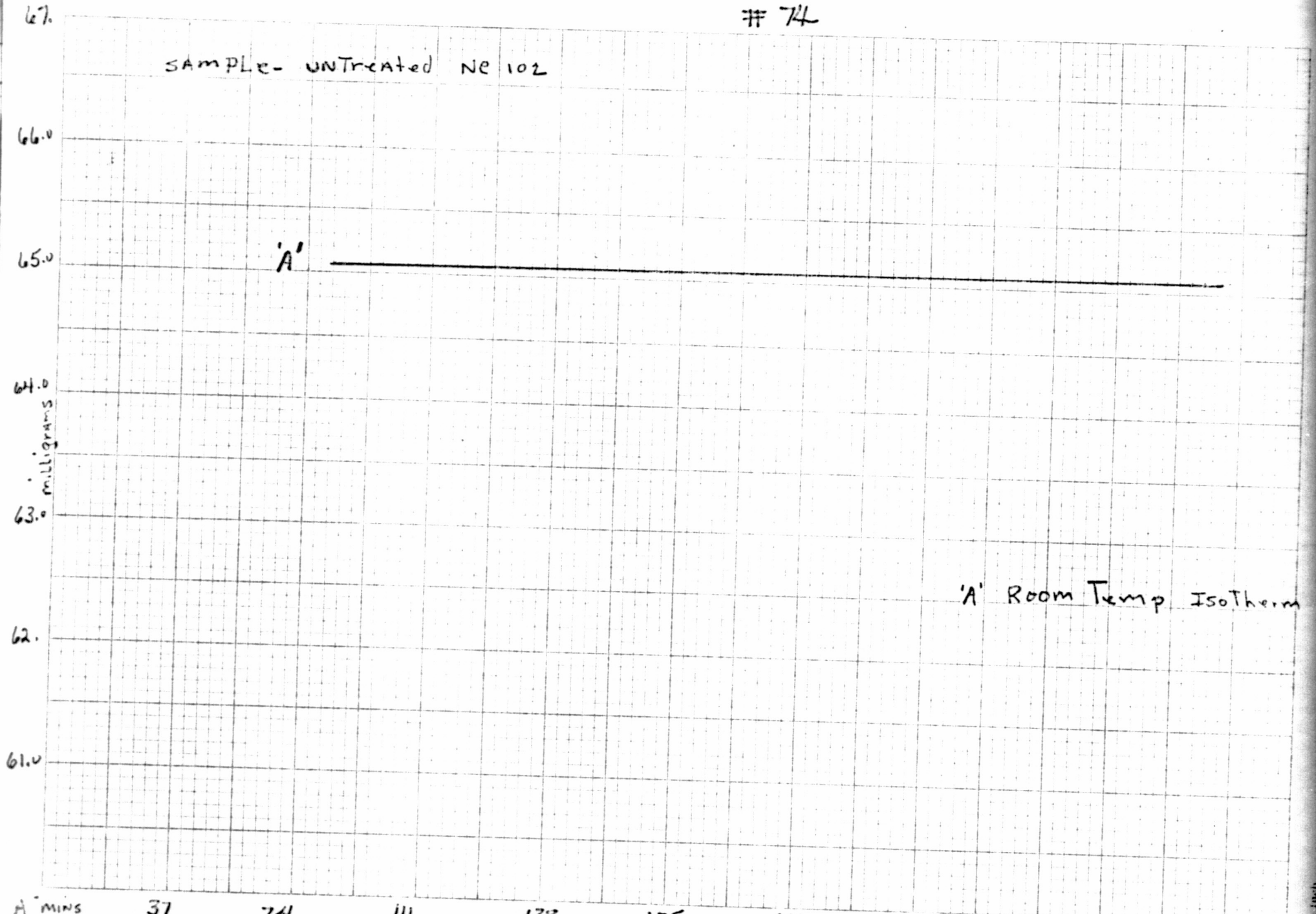
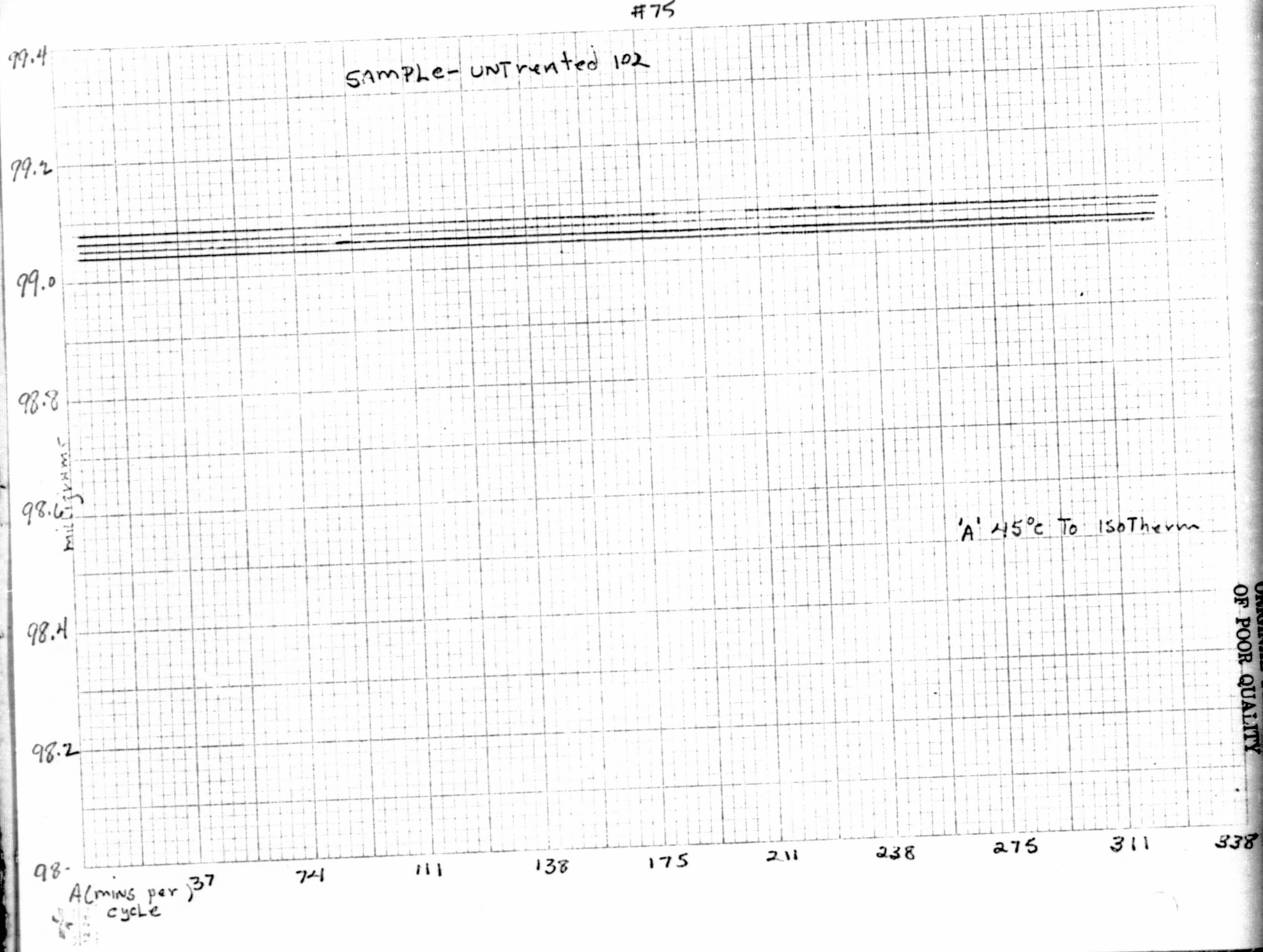

SAMPLE- UNTreated Ne 102
'A'
'A' Room Temp IsoTherm
67.
66.0
65.0
64.0
63.0
62.
61.0
milligrams
A' mins per cycle
37
74
111
138
175
211
238
275
311
338

#75



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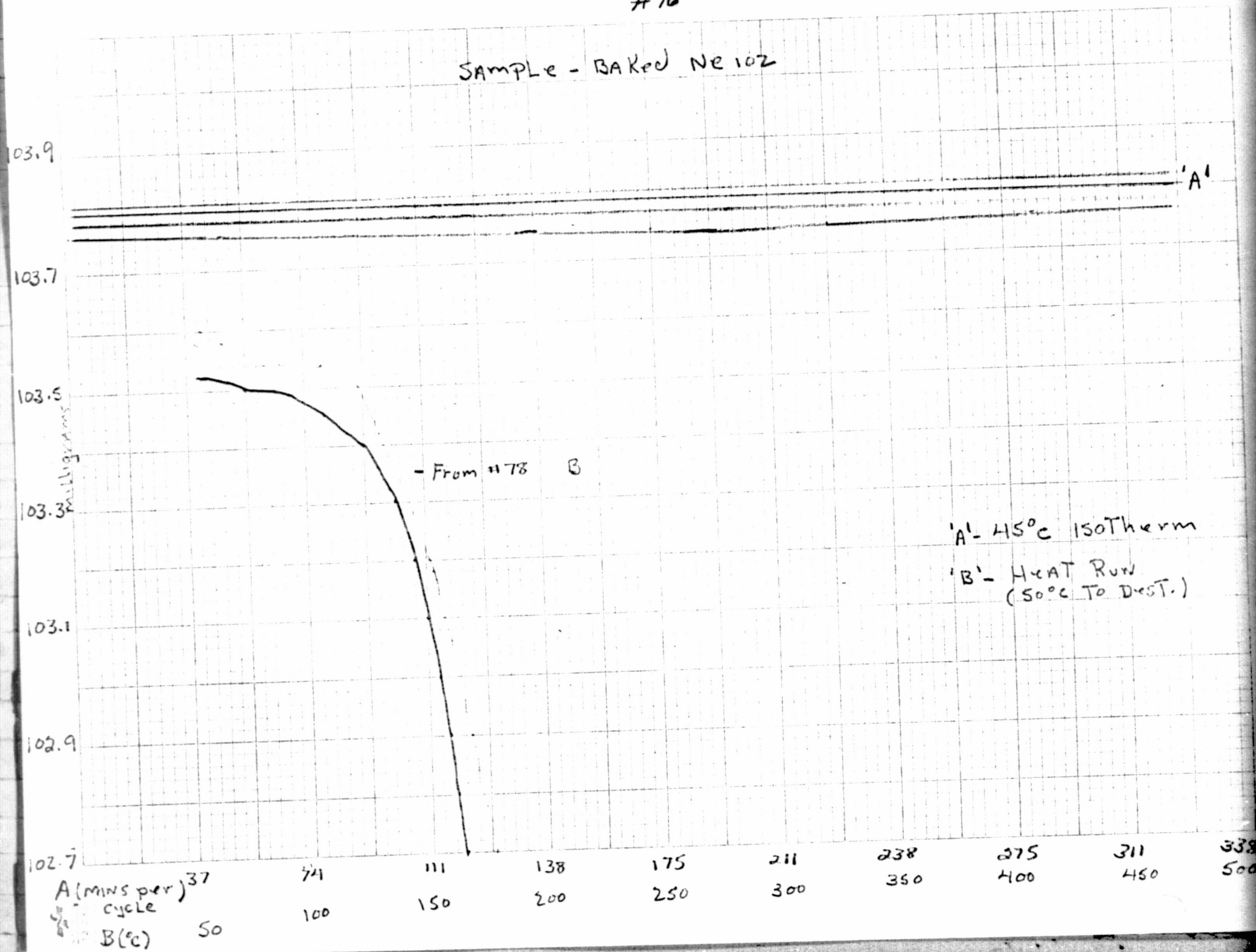

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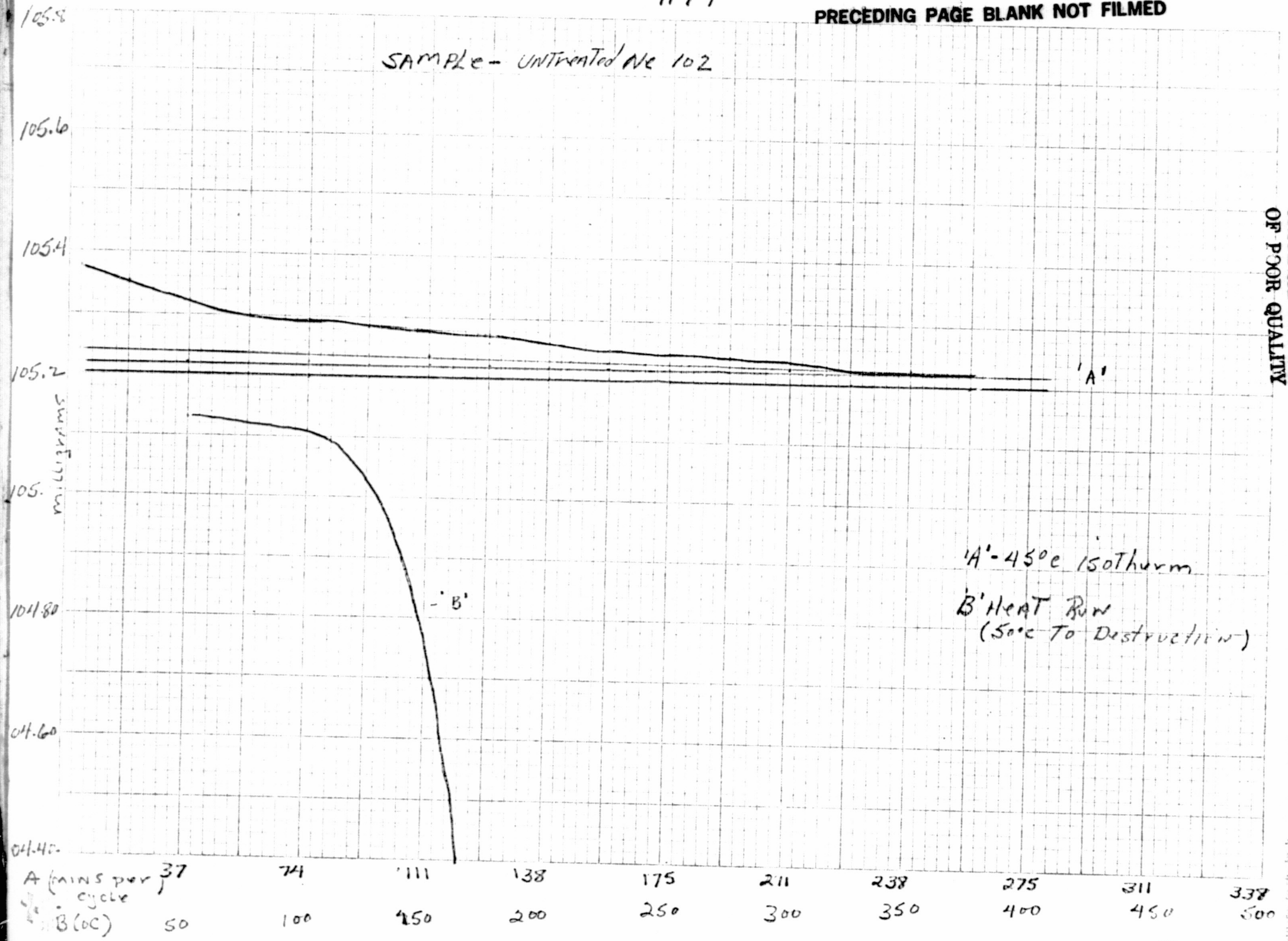

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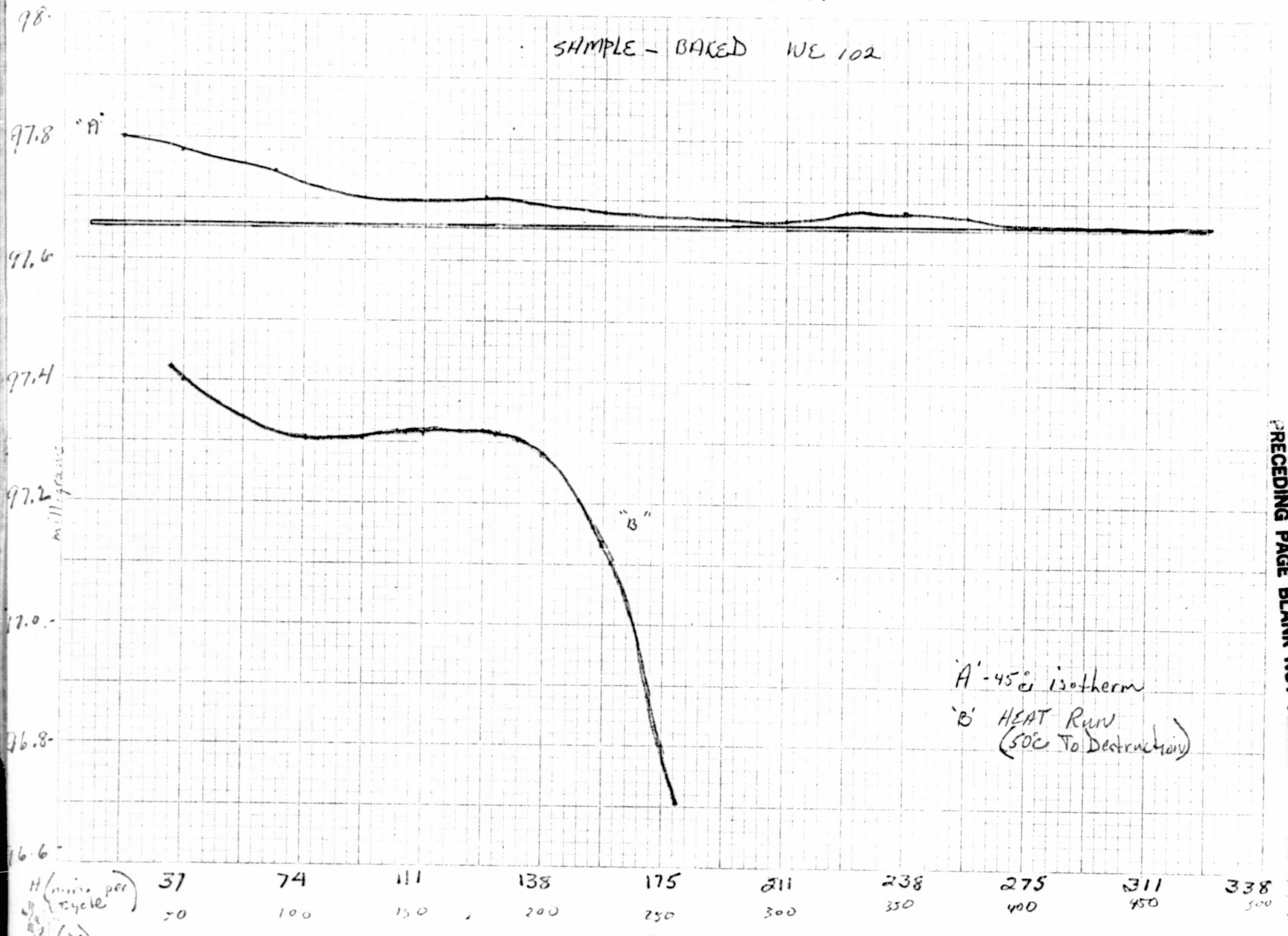

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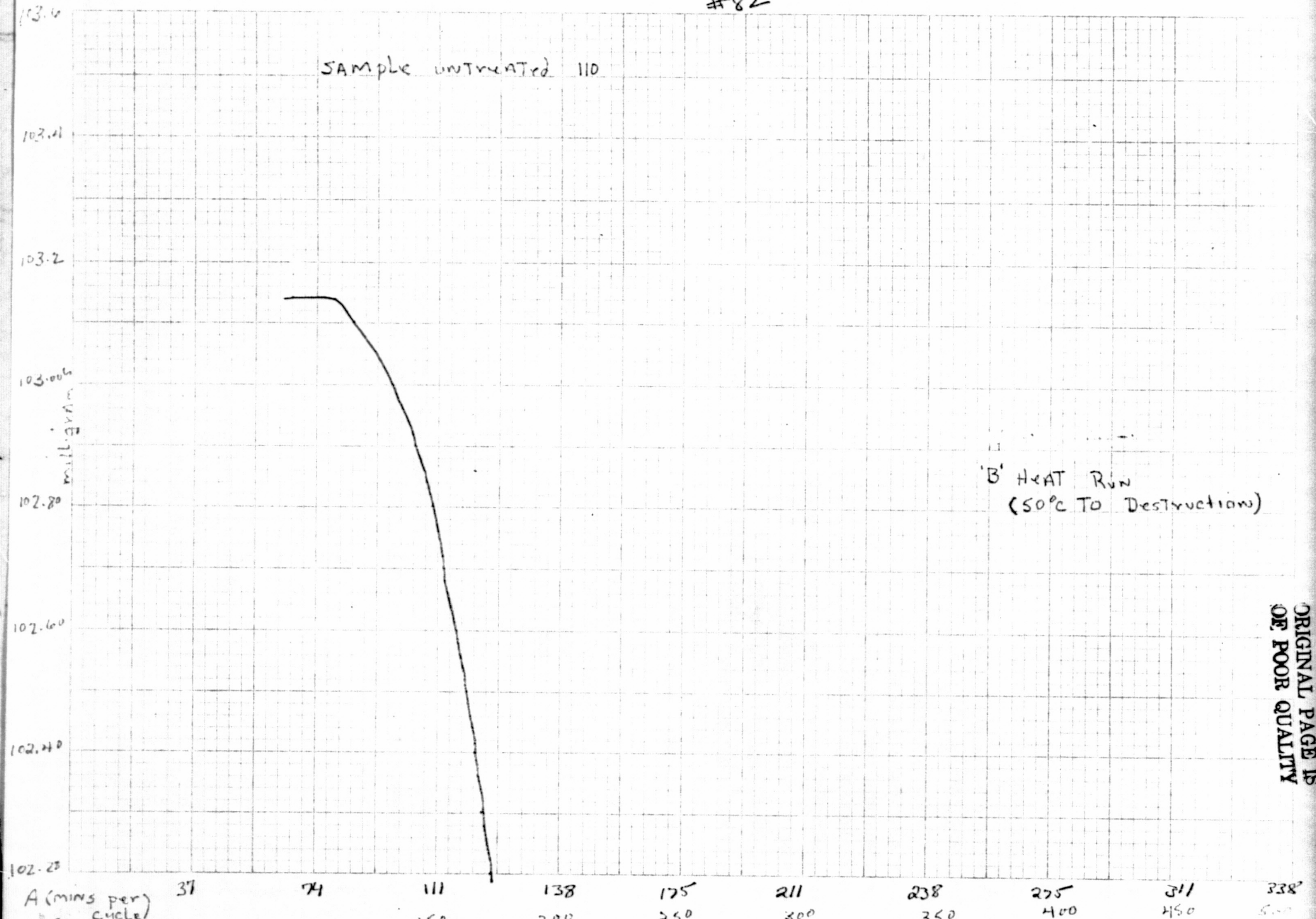

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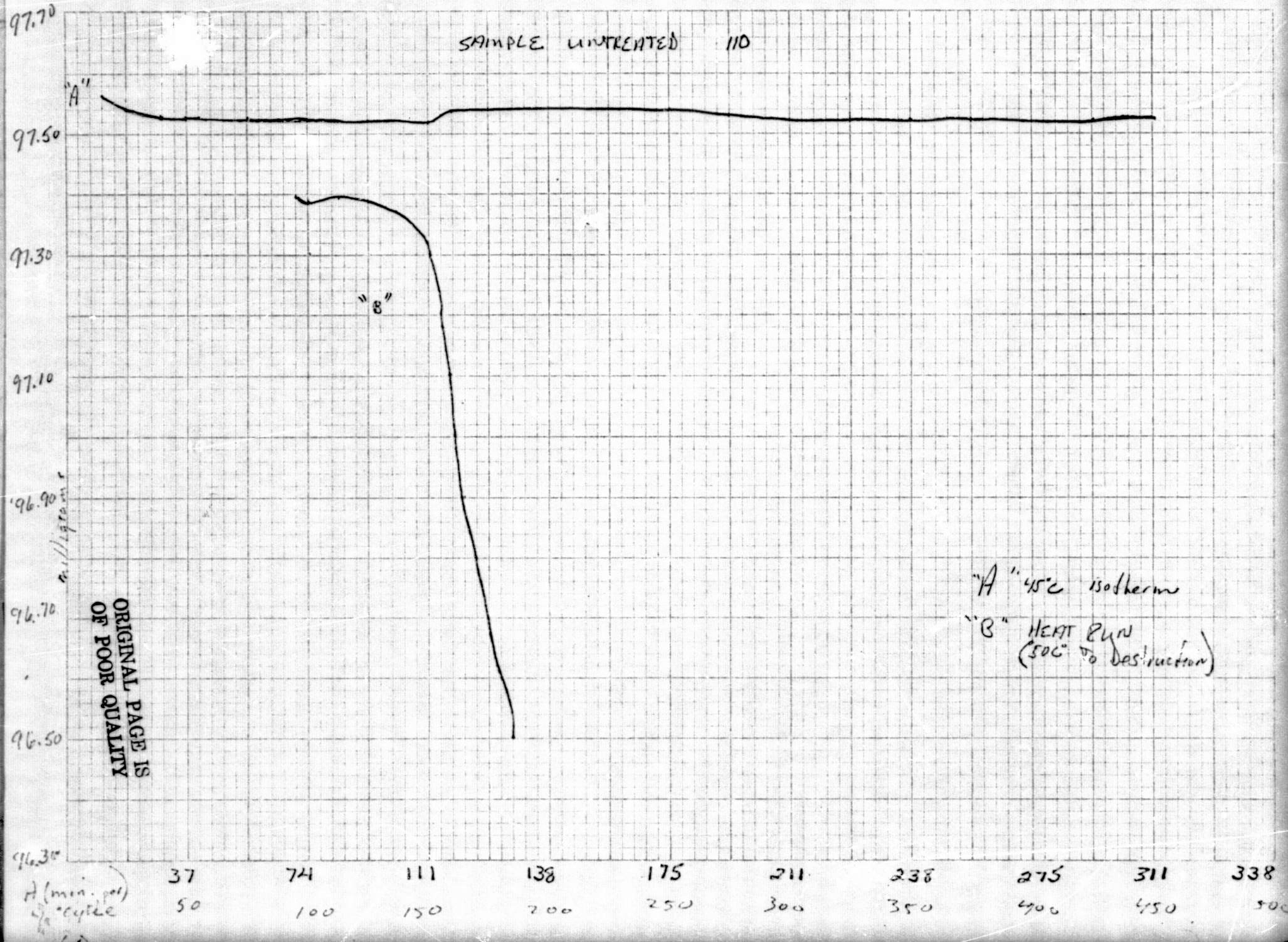

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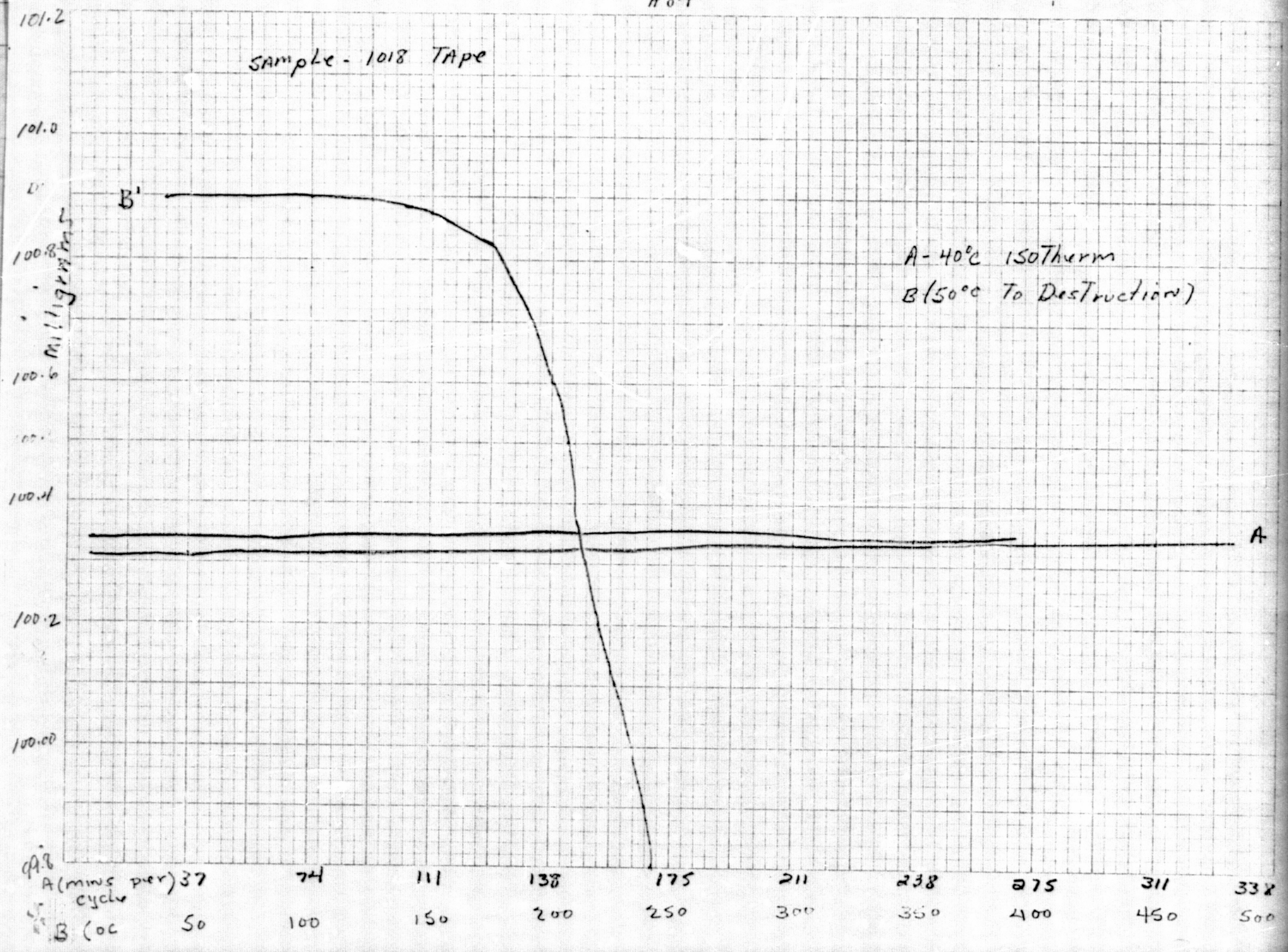
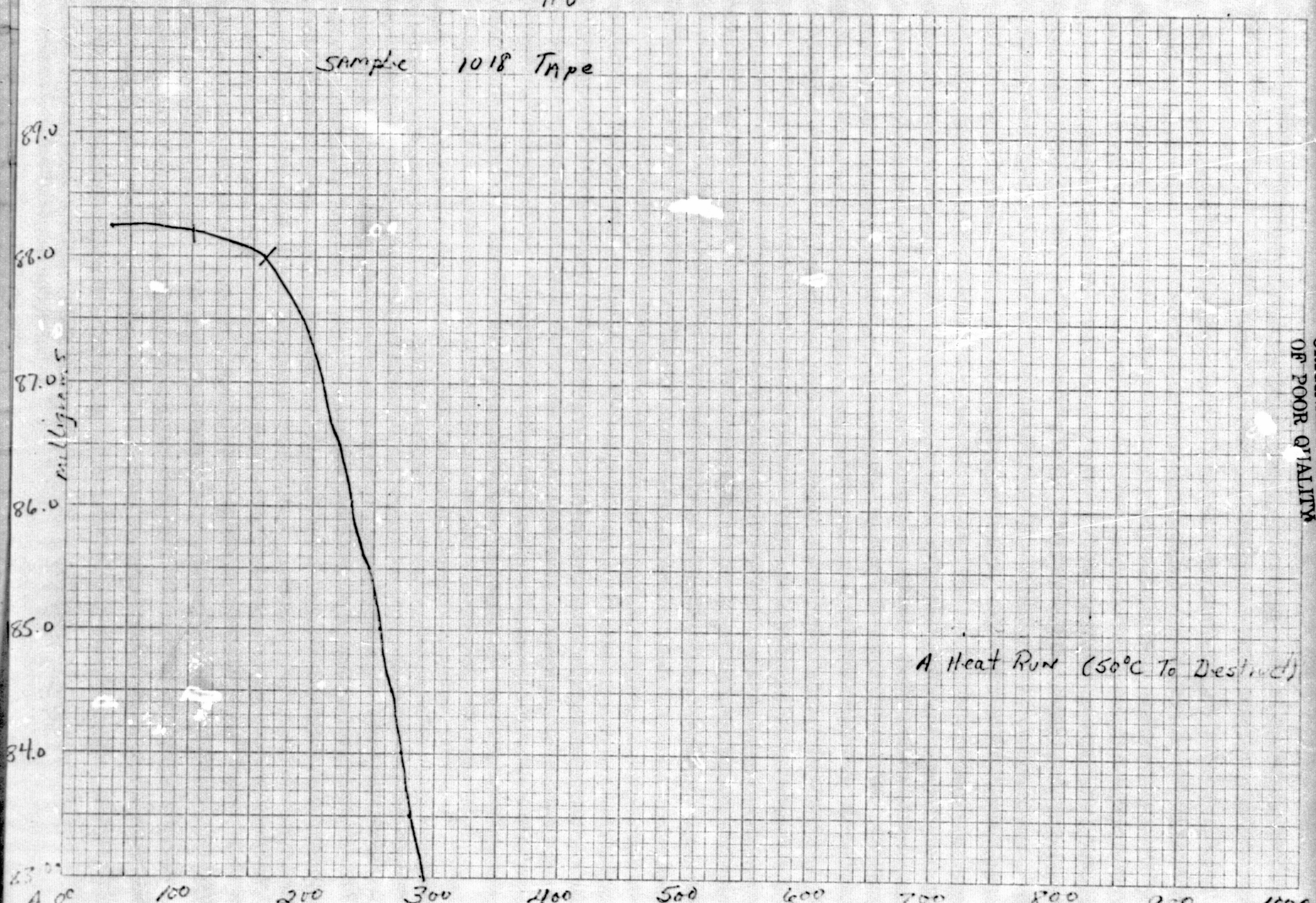

sample - 1018 Tape
A - 40°C Isotherm
B (50°C To Destruction)
B'
A
Milligrams
101.2
101.0
100.8
100.6
100.4
100.2
100.00
A (mins per cycle) 37 74 111 138 175 211 238 275 311 338
B (°C 50 100 150 200 250 300 350 400 450 500

7188



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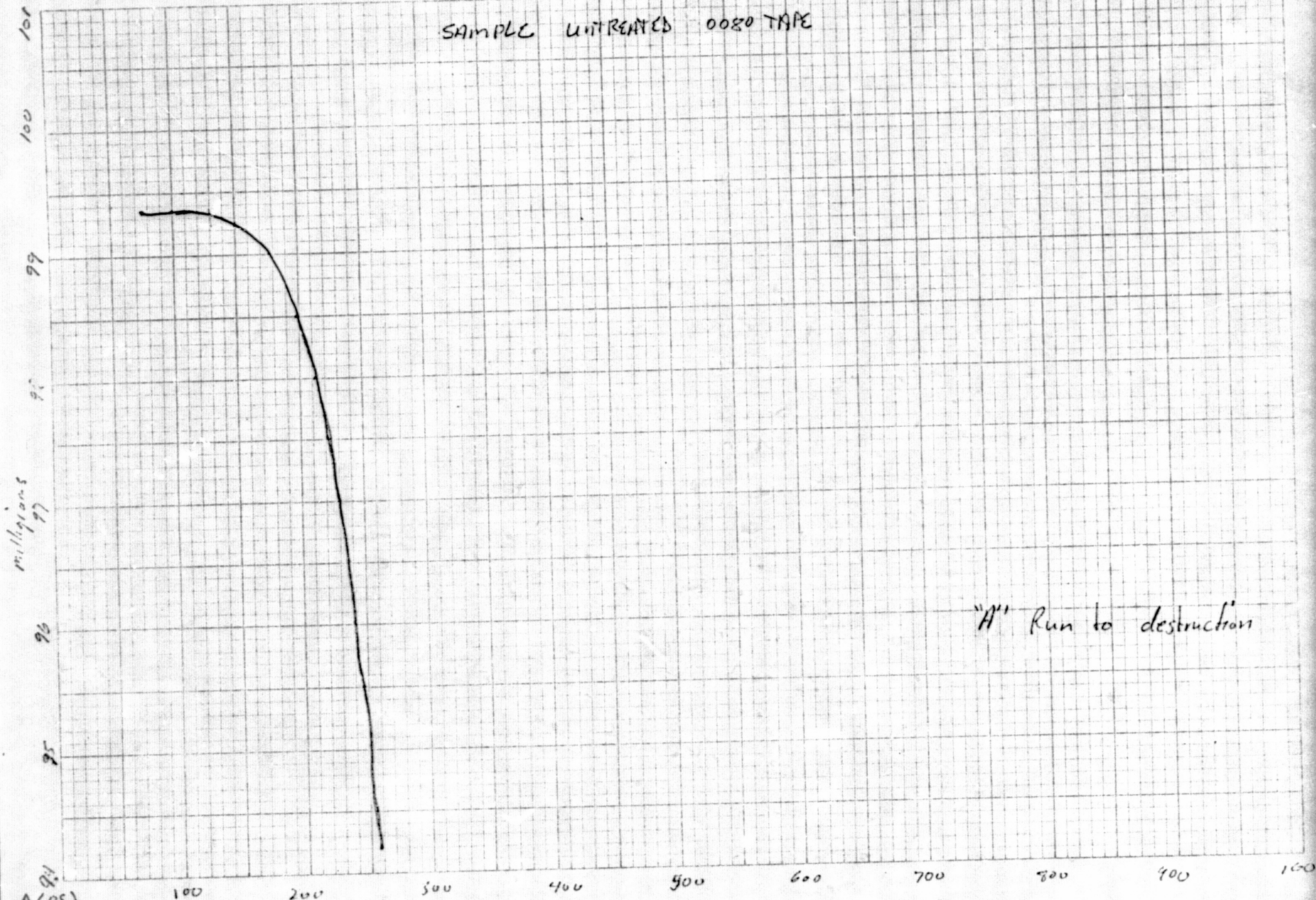
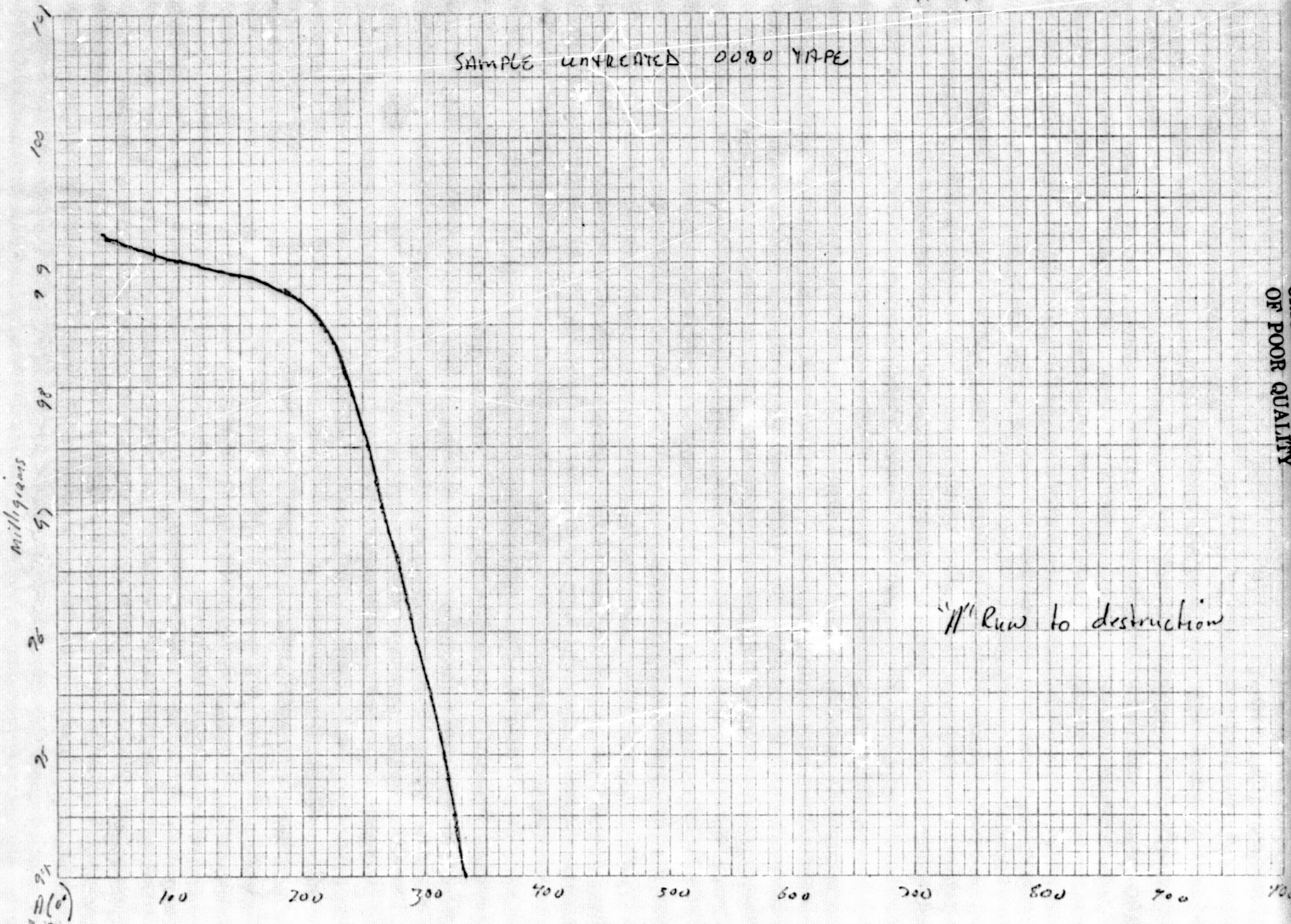
89

SAMPLE UNTREATED 0080 TAPE



"A" Run to destruction

#90



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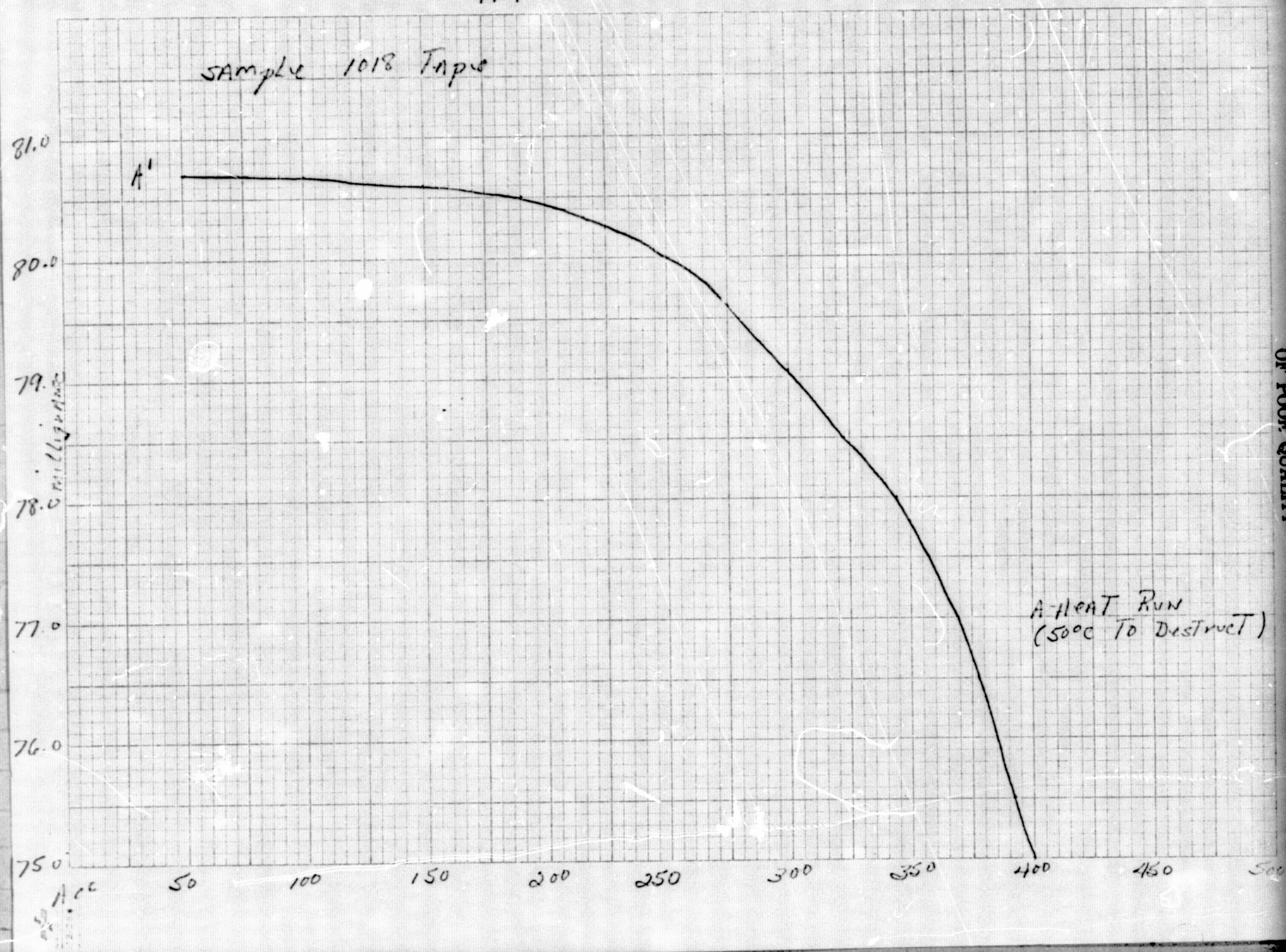

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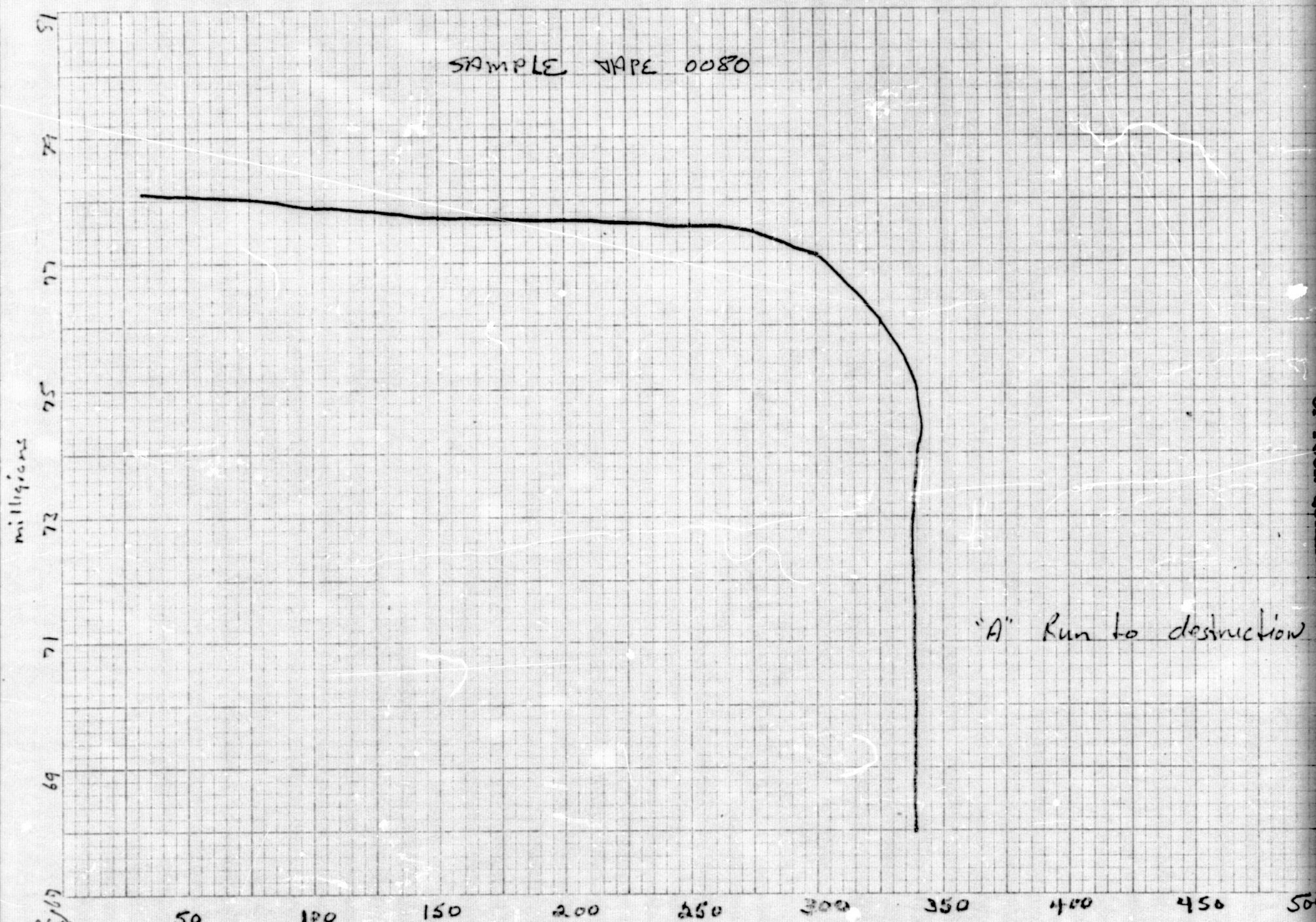
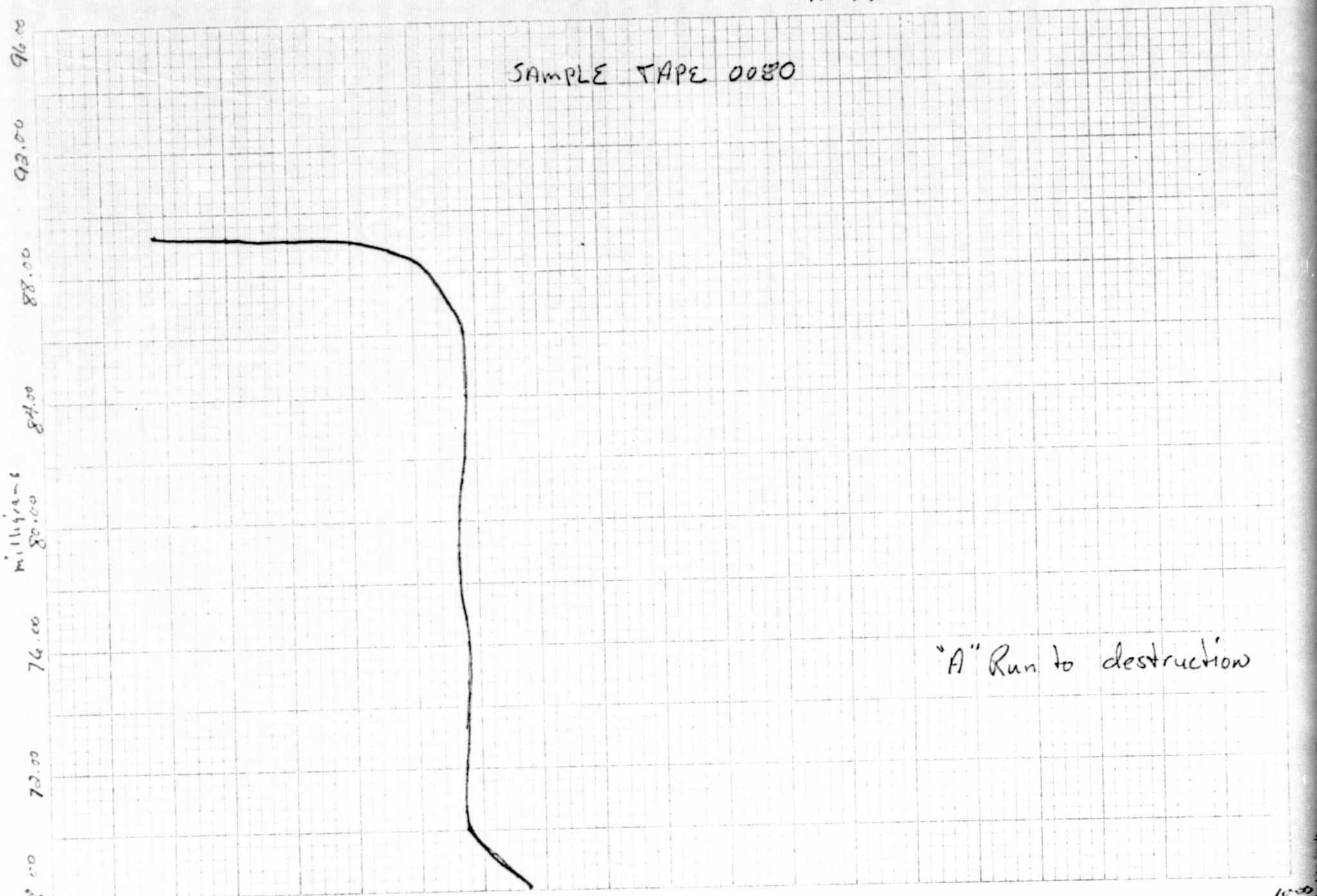
92


SAMPLE TAPE 0080
milligrams
51
73
77
75
73
71
69
50
100
150
200
250
300
350
400
450
"A" Run to destruction

#94

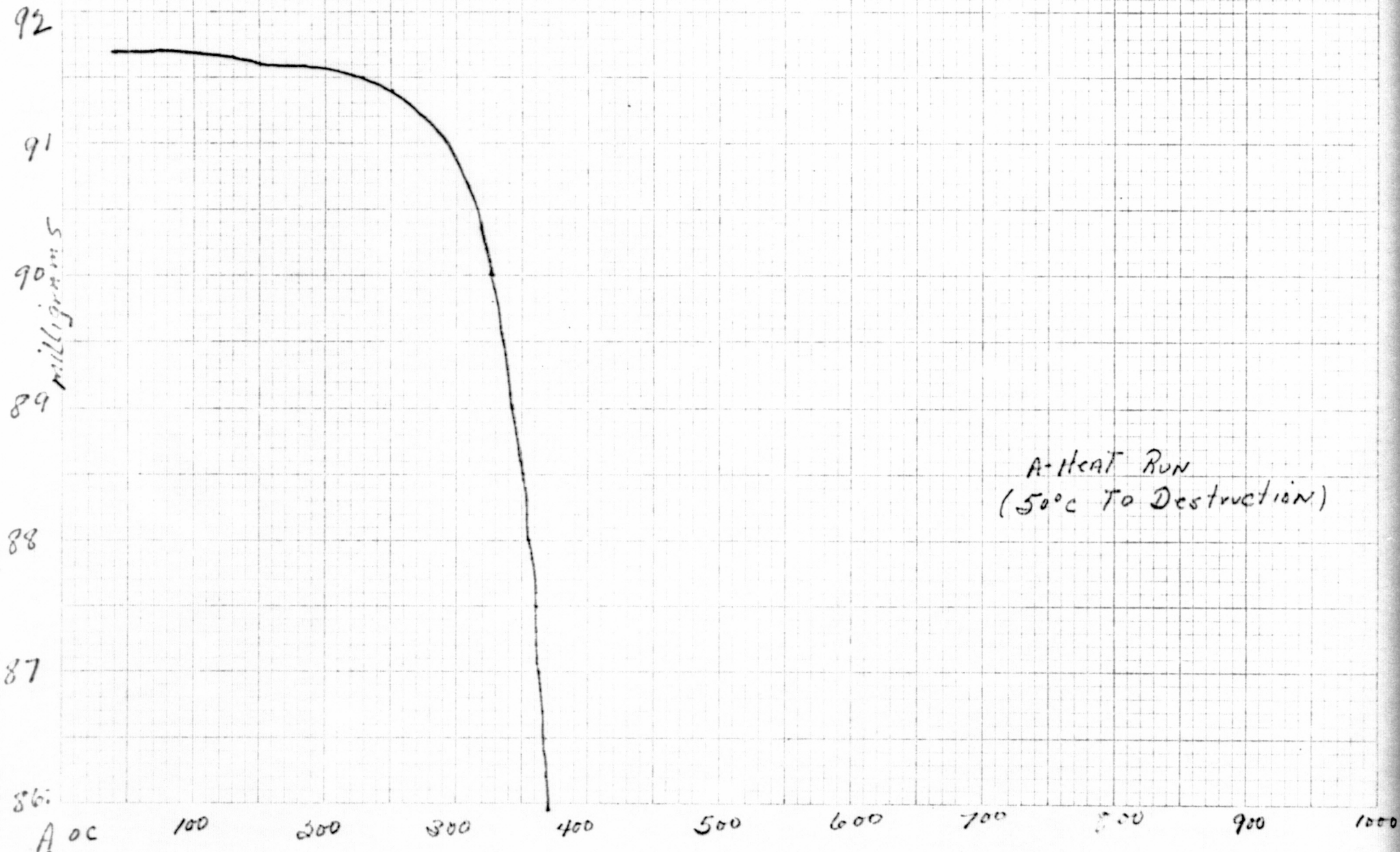
SAMPLE TAPE 0080



"A" Run to destruction

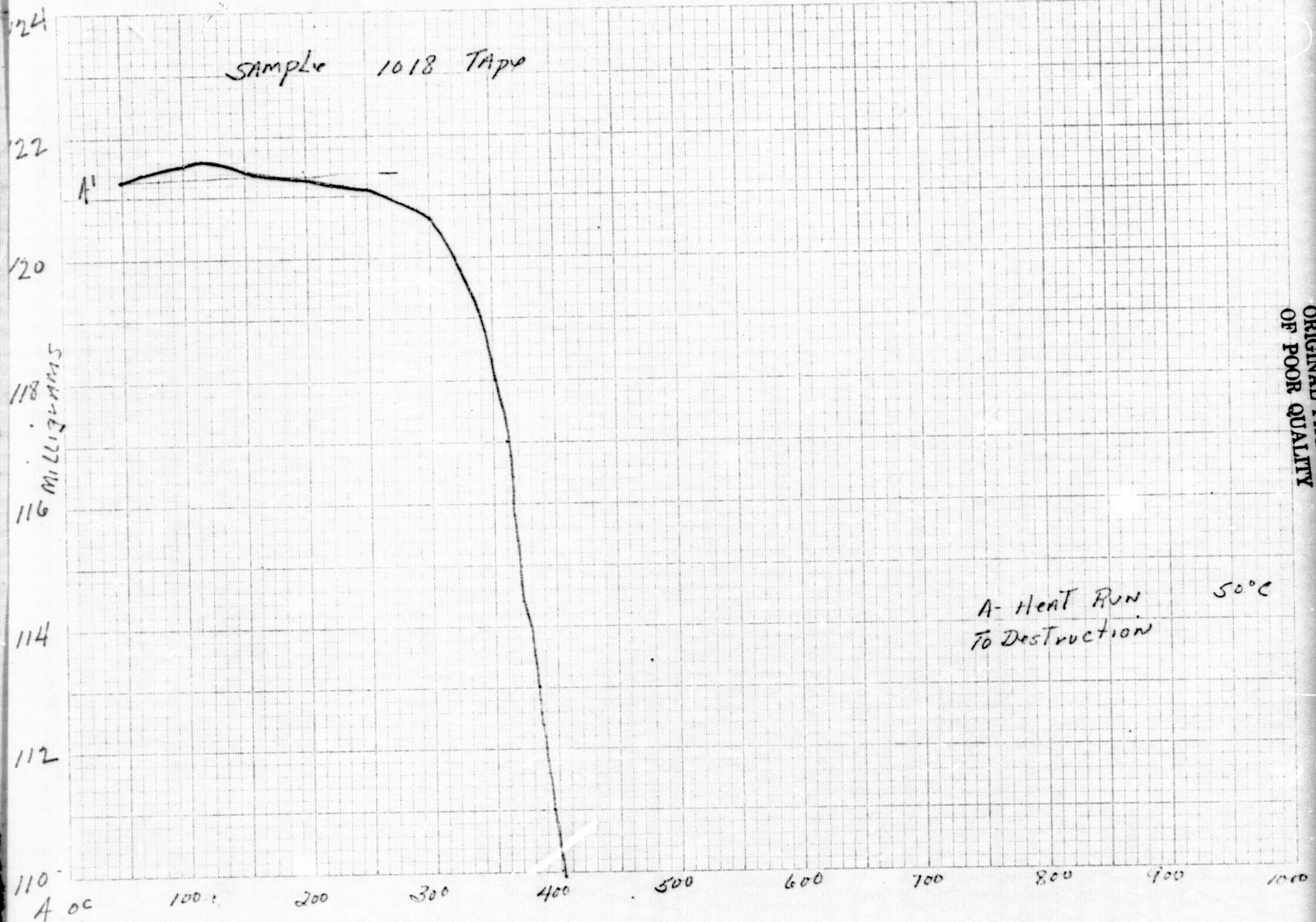
#95

SAMPLe- 1018 TAPe



A-HeAT RUN
(50°C To Destruction)

#96



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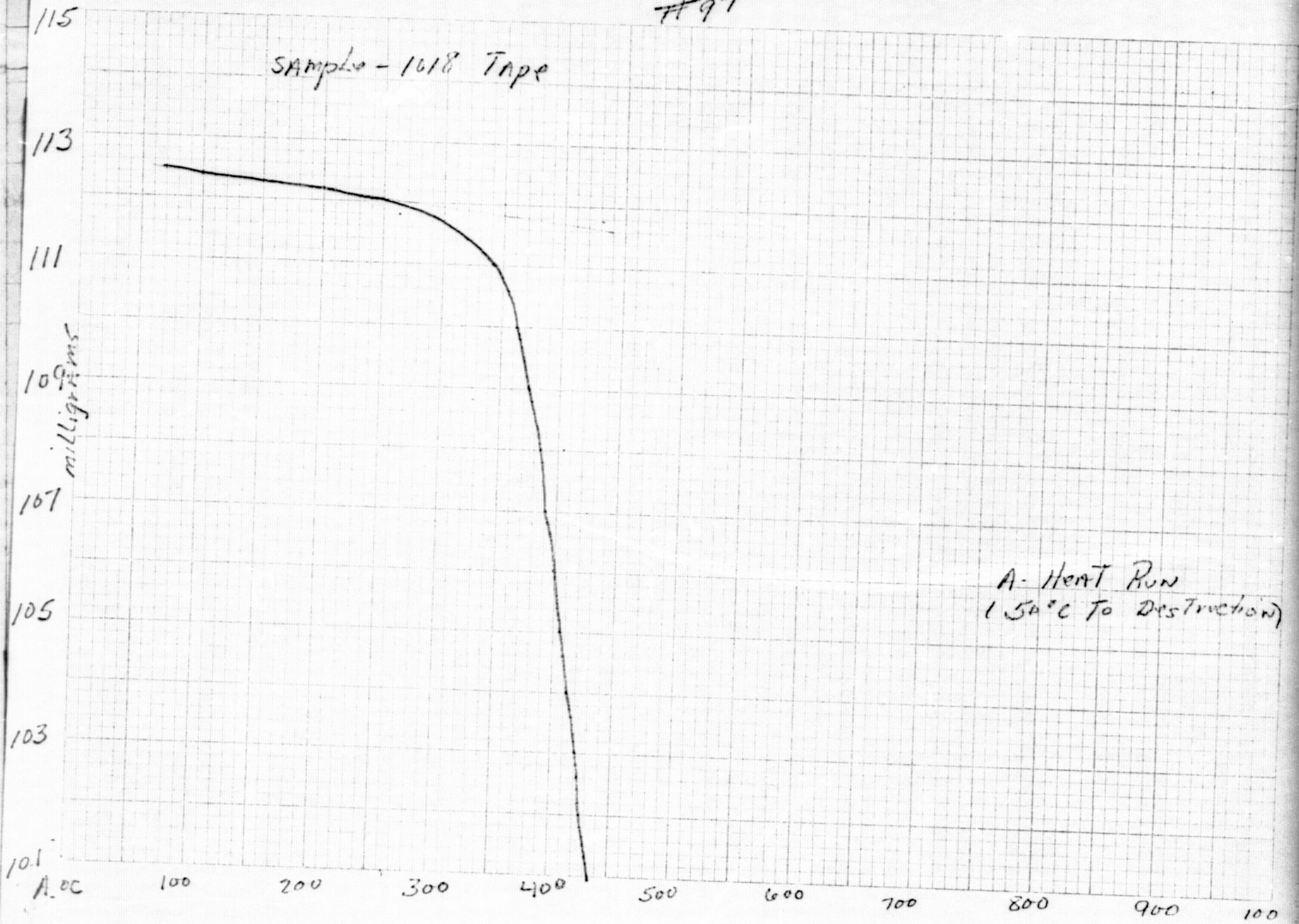
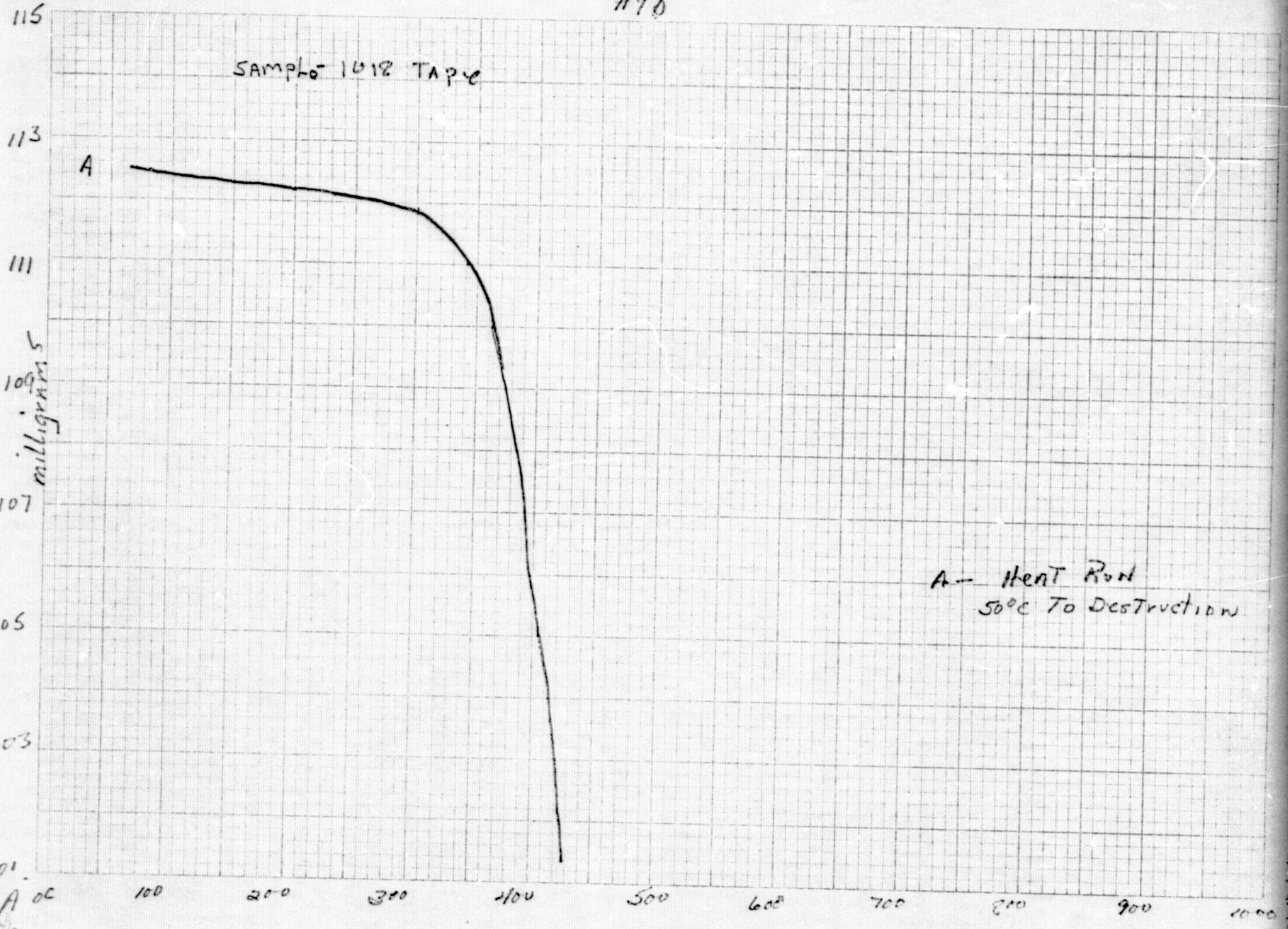
#97

Sample - 1618 Tape

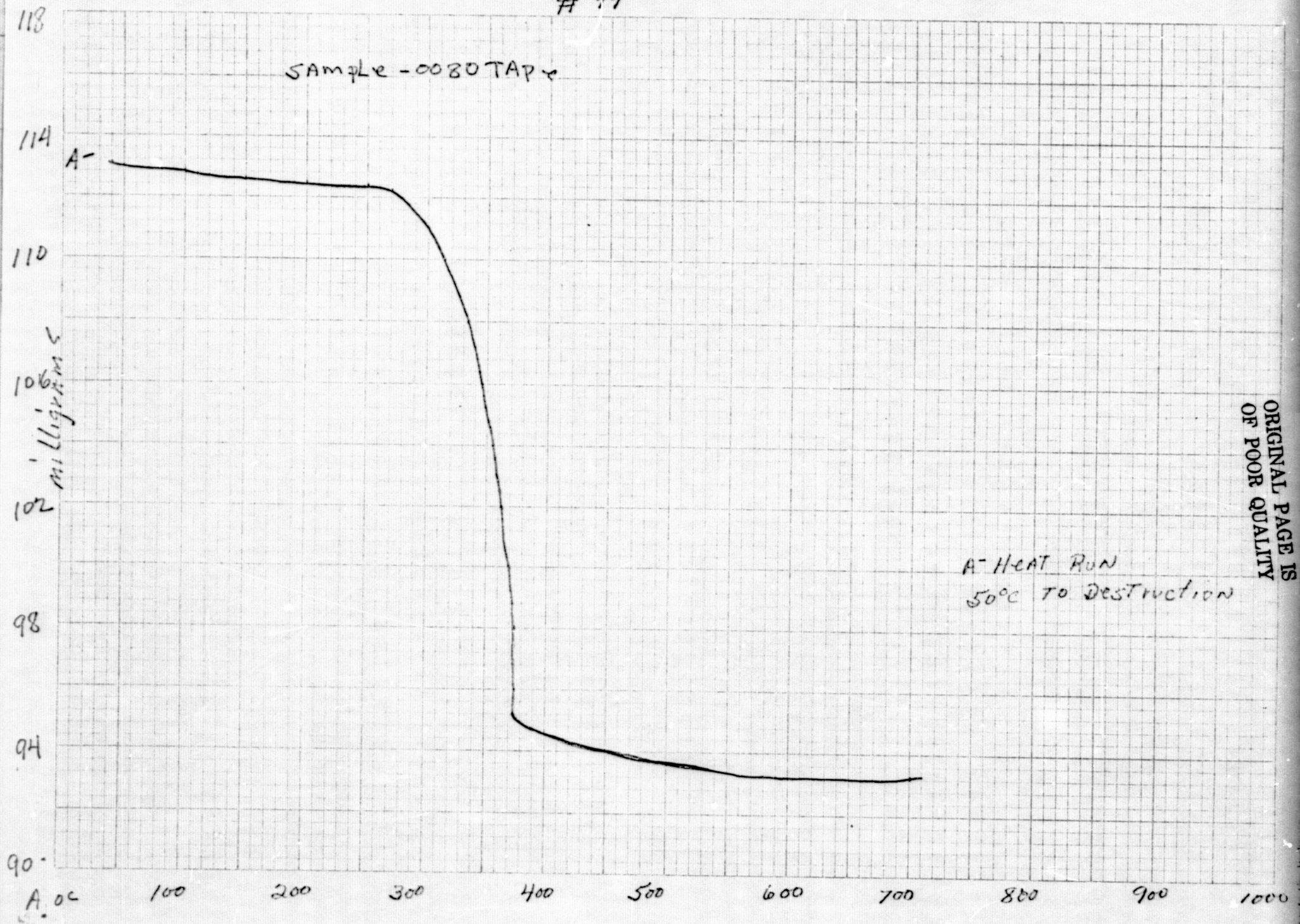


A - Heat Run
(150°C To Destruction)

#98


SAMPLE 1018 TAPE
A
115
113
111
109
107
105
103
101
milligrams
A — HEAT RUN
50°C TO DESTRUCTION
A °C
100
200
300
400
500
600
700
800
900
1000

99



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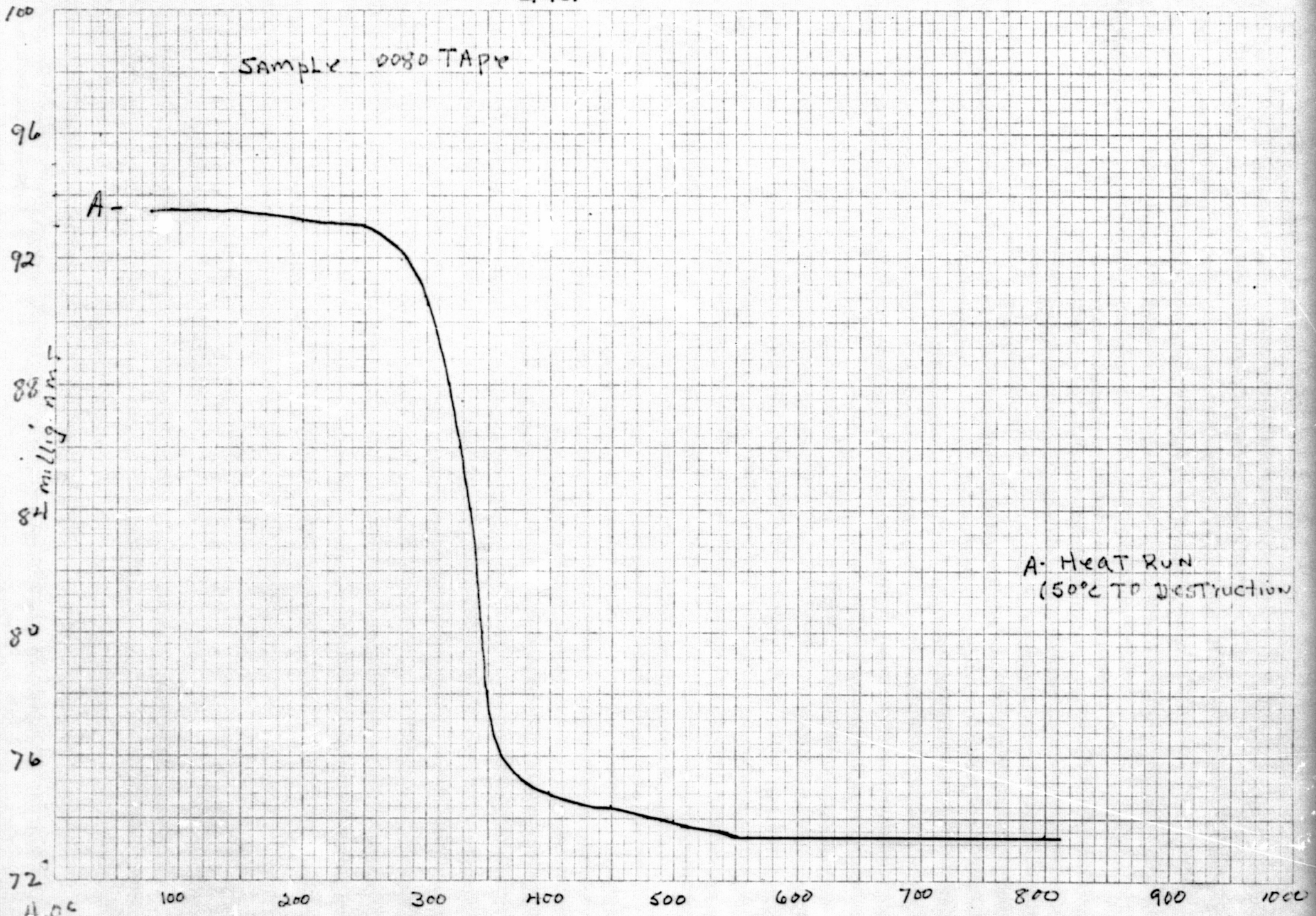
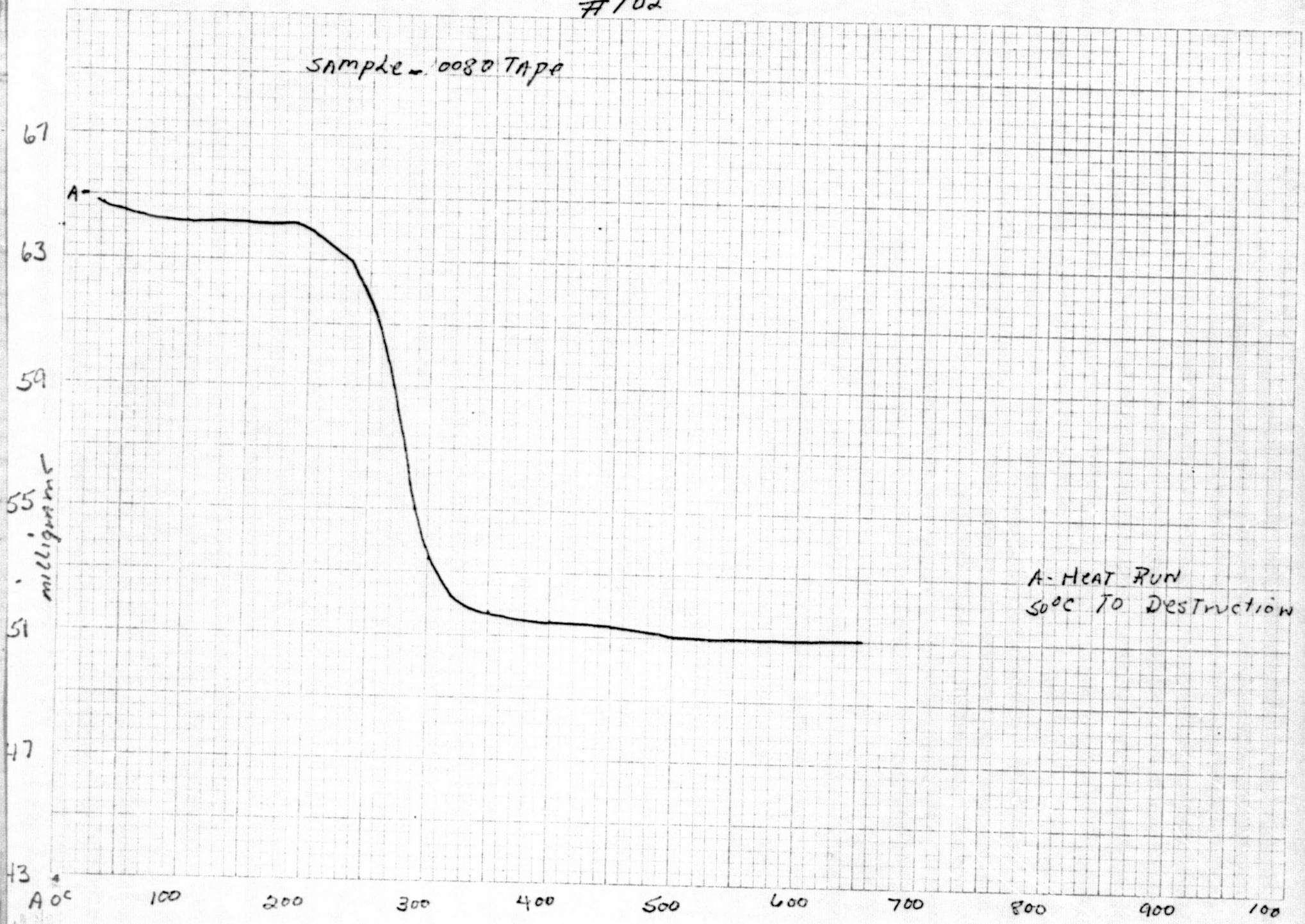
#101

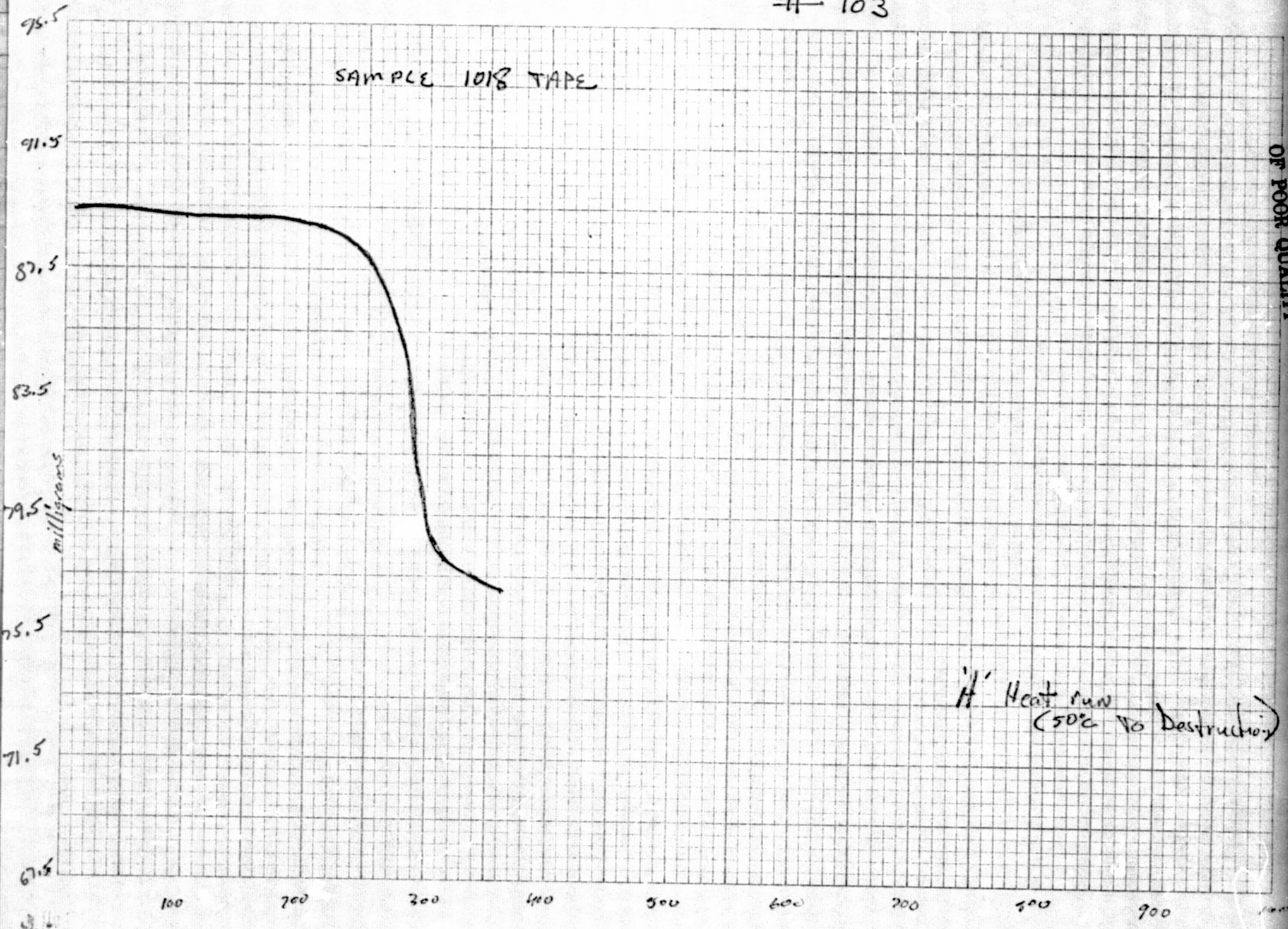

SAMPLE 0080 TAPE
A-
A. HEAT RUN
150°C TO DESTRUCTION
100
96
92
88
84
80
76
72
milligrams
100
200
300
400
500
600
700
800
900
1000
A.0C

#102

sample - 0080 Tape



103



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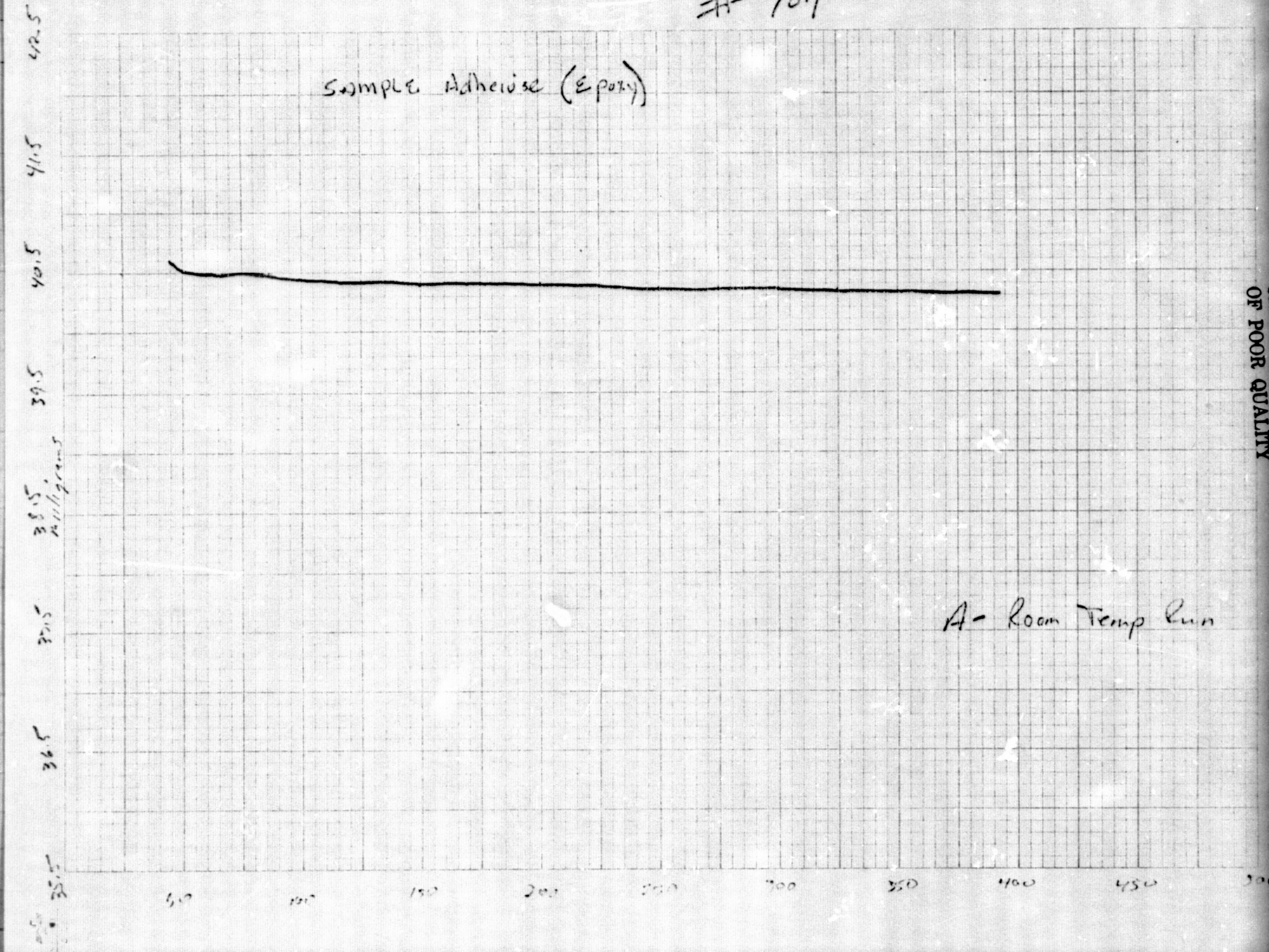
#106

SAMPLE Black graphite cloth

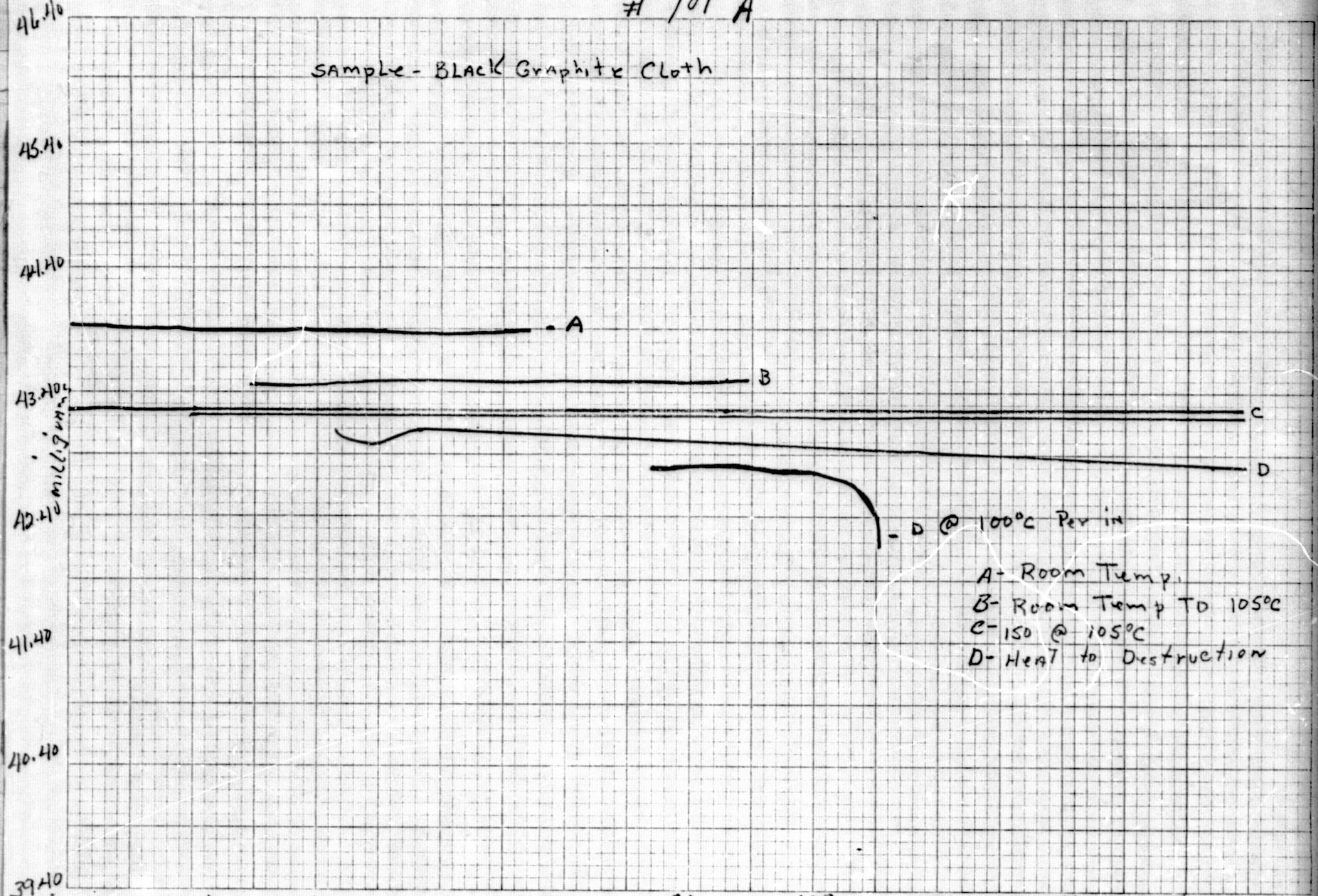
'A' isothermal 105°
'B' rerun of above
'C' 150 @ 125°C

#107



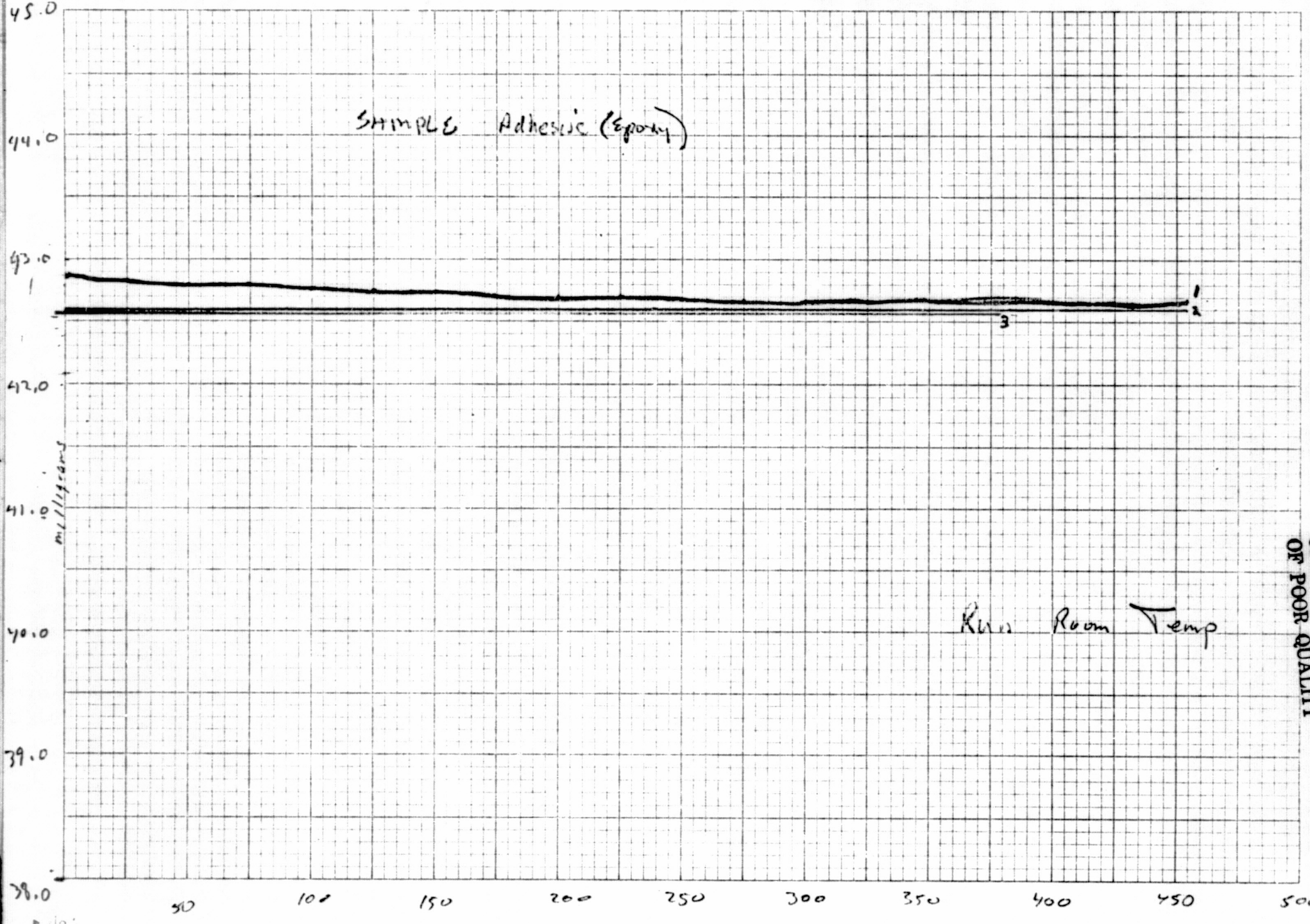
OF POOR QUALITY

#107 A



A(mins per cycle)		36	54	72	90	108	126	142	150	168
B(°C)	20	40	60	80	100	120	140	160	180	200
C(MPC)	37	74	111	138	175	211	238	275	311	338
D°C)	50	100	150	200	250	300	350	400	450	500

#108



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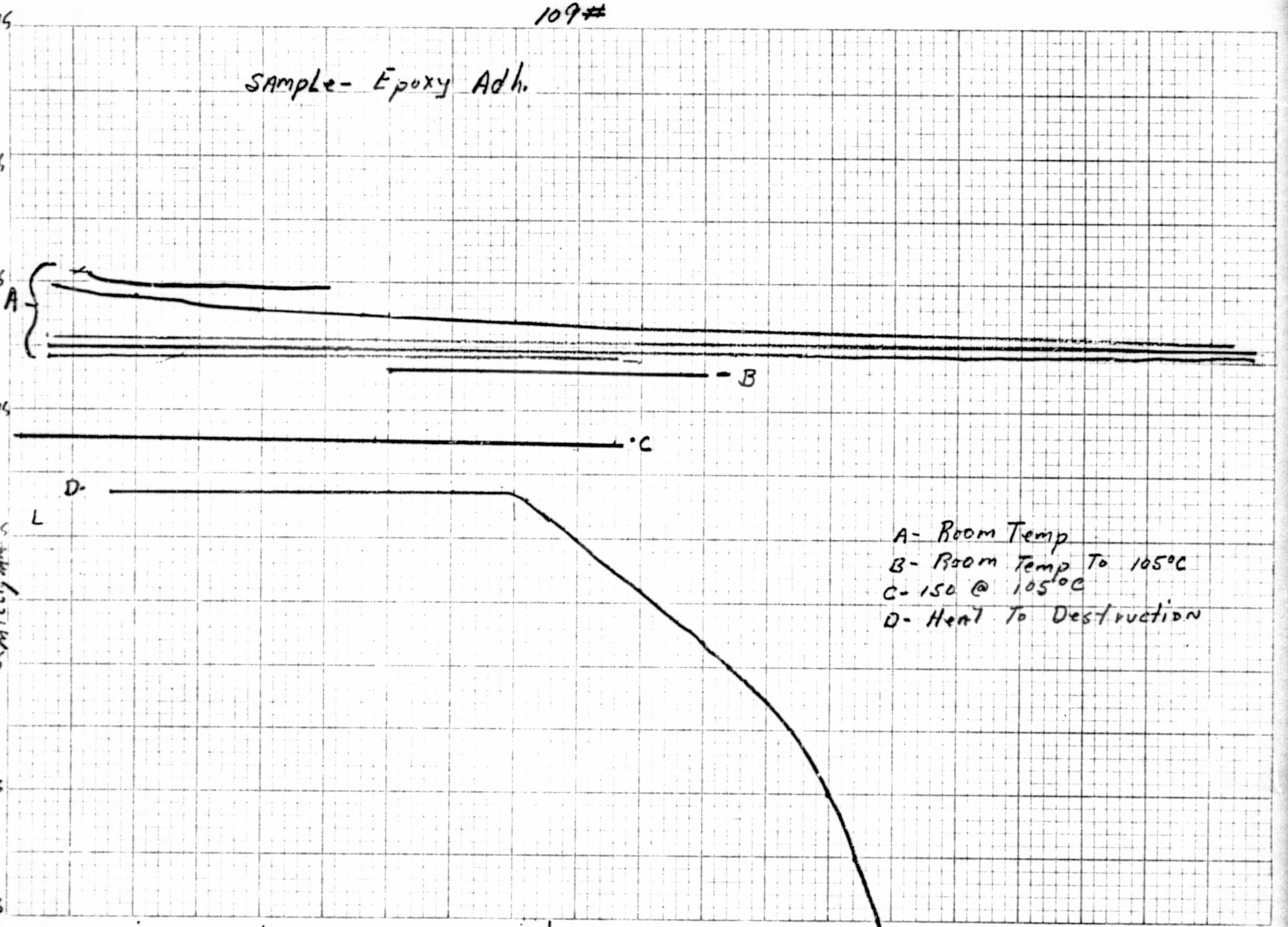
10 Squares to the Inch

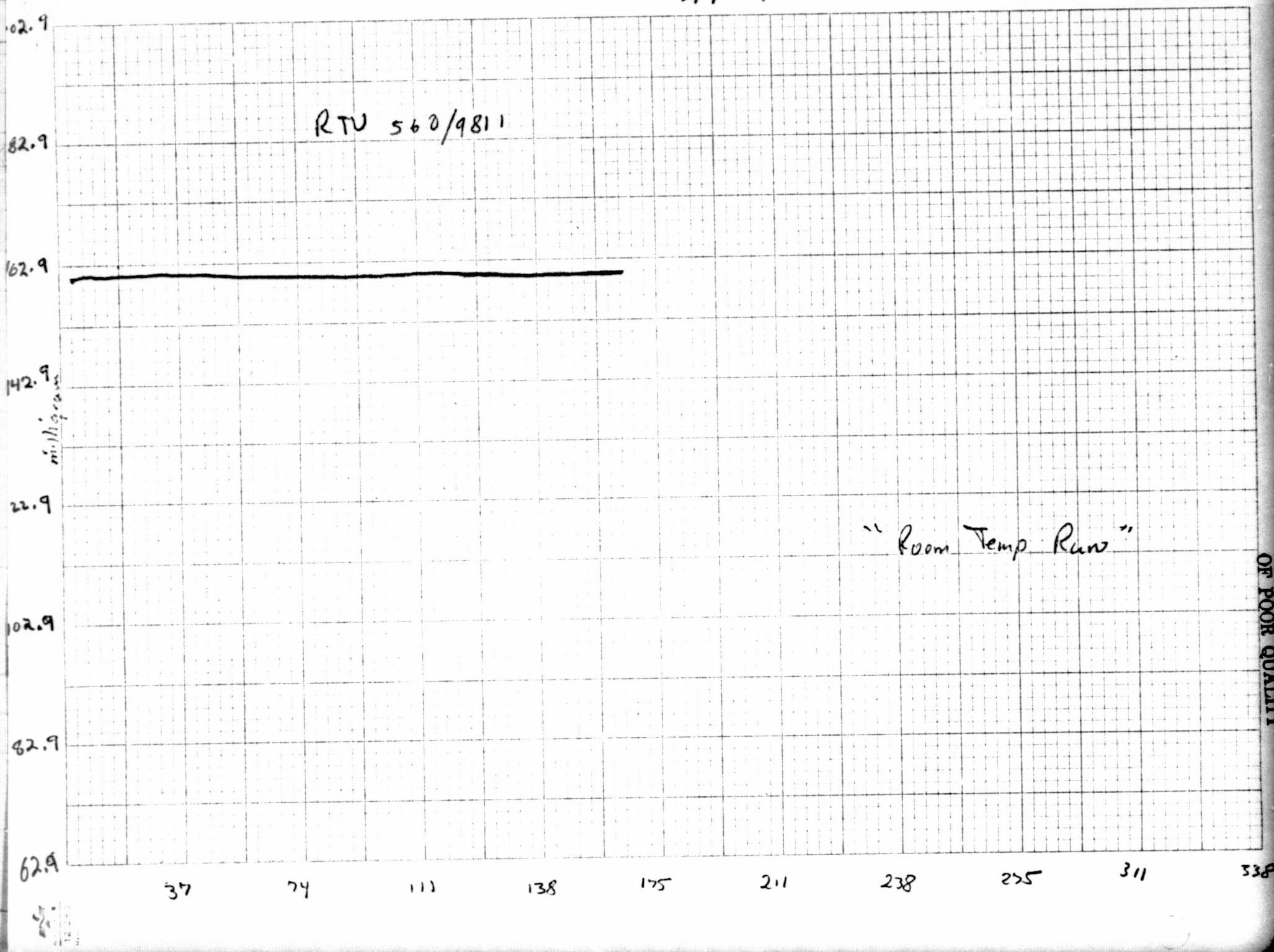
109#

Sample- Epoxy Adh.



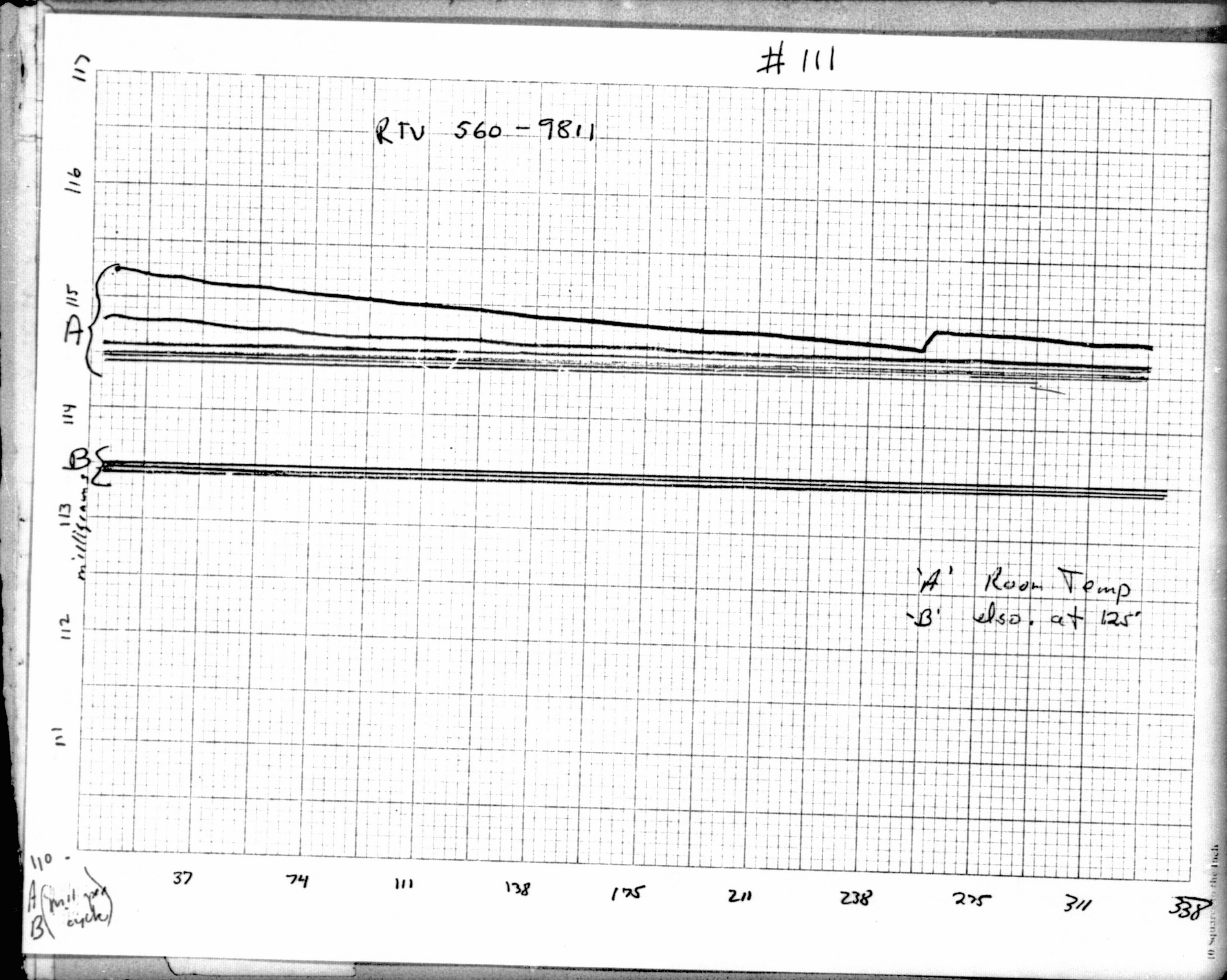
A(mins per cycle)	37	74	111	138	175	211	238	275	311	338
B °C	20	40	60	80	100	120	140	160	180	200
C (mpc)	37	74	111	138	175	211	238	275	311	338
							350	400	450	500

#110

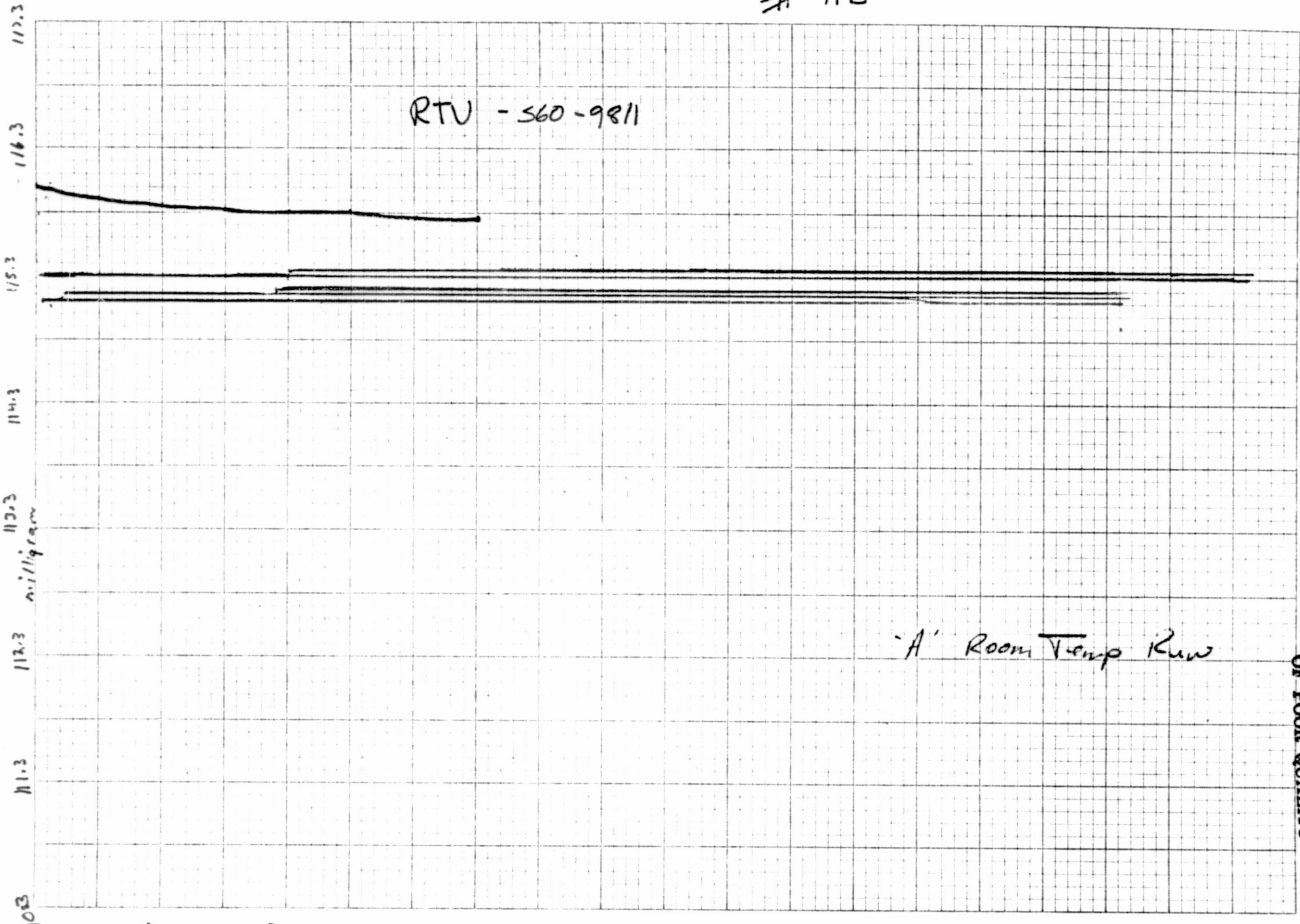


OF POOR QUALITY

#111


RTV 560-9811
117
116
115
114
113
112
111
110
milligrams
A
B
'A' Room Temp
'B' also. at 125°
37
74
111
138
175
211
238
275
311
338
A (mil per cycle)
B

#112



(3.5 per cycle)

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DIFFERENTIAL THERMAL ANALYSIS (DTA)

Differential thermal analyses are conducted with a DuPont Model 900 DTA unit. Two tubes, one containing sample and the other a reference material, such as glass beads, are heated at a uniform rate in a heating block. The sample and the reference material will initially be at the same temperature. The temperature differential will remain zero as the two are heated unless the sample undergoes an endothermic or exothermic reaction. The DTA unit plots the temperature of the heating block on the x-axis. On the y-axis it plots the difference in temperature between the sample and reference, ΔT. An exotherm is plotted as a rise from the base line, an endotherm as a decrease from the base line.

Comparison of graphs 18 and 19 shows that a large part of the apparent slow endotherm on C-19 alone is due to an instrumental thermocouple imbalance. In samples of C-19 with excess 40% KOH this imbalance is masked by the high specific heat of the solution.

Several silicone rubbers were analyzed by DTA, these being NASA Goddard #AF-E-332 silicone, S-383-70 silicone, and E-515-80(1). The temperature range was from -125° C to 25° C. The samples, plus the heating block which they were in, were cooled in liquid nitrogen and then warmed to room temperature at a programmed rate.

CONCLUSIONS

C-19 does not undergo either an endothermic or an exothermic reaction at temperatures below 100° C. It does not react alone, nor with excess 40% KOH, nor with excess Ag_2O in 40% KOH.

CONTINUATION

Additional runs are being made with other separator materials of interest to NASA.

EXPERIMENTAL PROCEDURE

Sample C-19, a cellophane separator used in batteries, was tested. The separator has been impregnated with Ag_2O and is used in a KOH electrolyte.

Glass beads are used as the reference. Runs were made of 40% KOH solution against glass beads to determine any endotherms caused by evaporation of water.

Runs were made of glass beads vs. glass beads to detect any slope in the base line caused by thermocouple imbalance.

DISCUSSION OF RESULTS

The C-19 in 40% KOH showed no reaction below 115° C. Between 115° C and 120° C there is a sharp endotherm. If the sample is cooled and reheated the endotherm does not occur. The same results are found for C-19 plus Ag_2O in 40% KOH (see graphs 10-1 and 11).

The C-19 alone showed a much less sharp endotherm starting at about 90° C. (See graphs 13, 15). A sample of C-19 stored over "Drierite" showed a similar diffuse endotherm with a less rapid drop between 90° and 110° C (See graph 14, 17).

The samples of silicone rubber, graphs #60, 61, 62, showed no phase changes in the range -125° C to 25° C. On the initial samples a phase change for condensed water was shown at about 0° C. The latter sample tubes (#60, 61, 62) were closed and showed no ice to water phase change. (See graphs 60, 61, 62)

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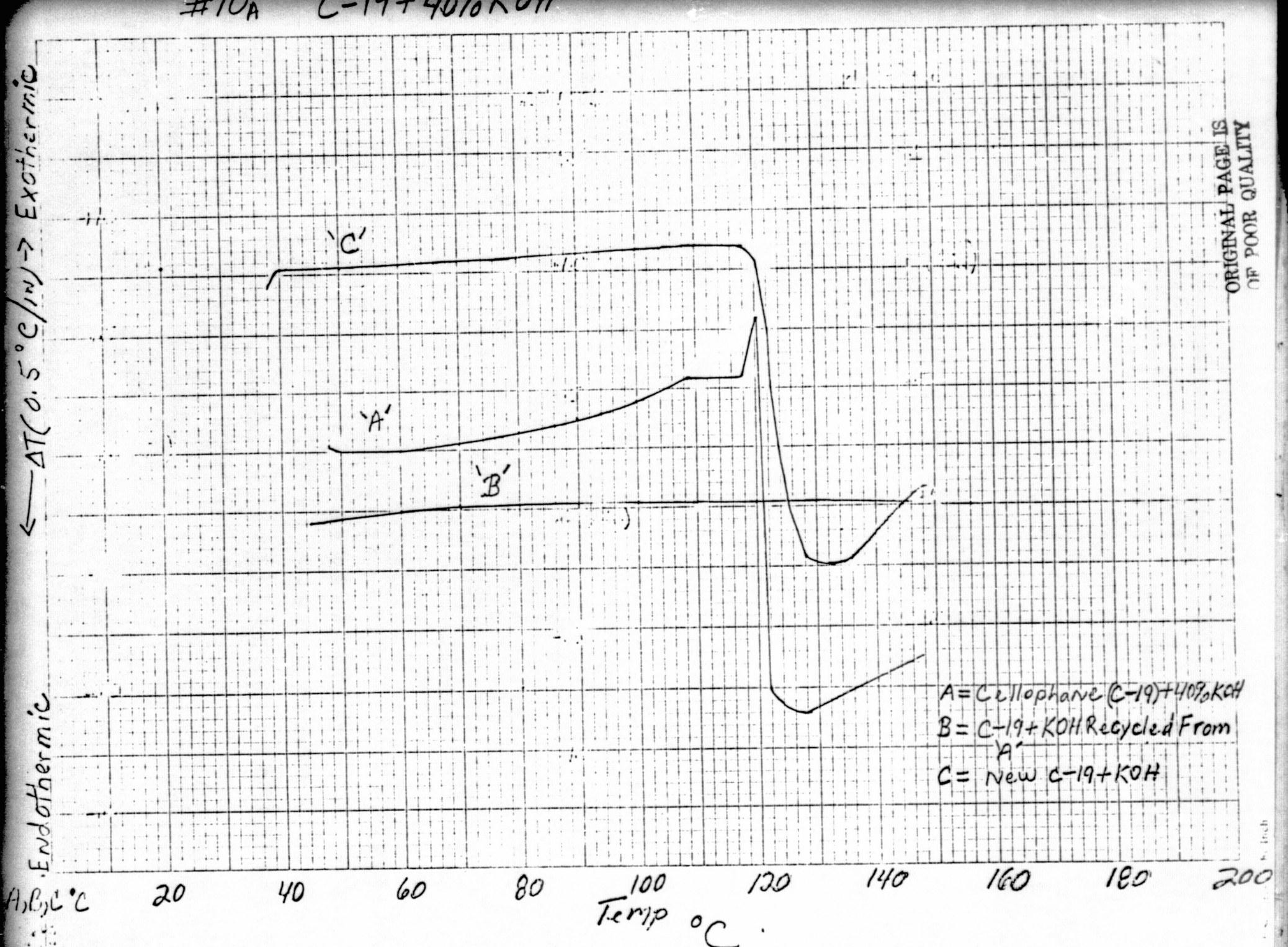
#10A C-19+40% KOH



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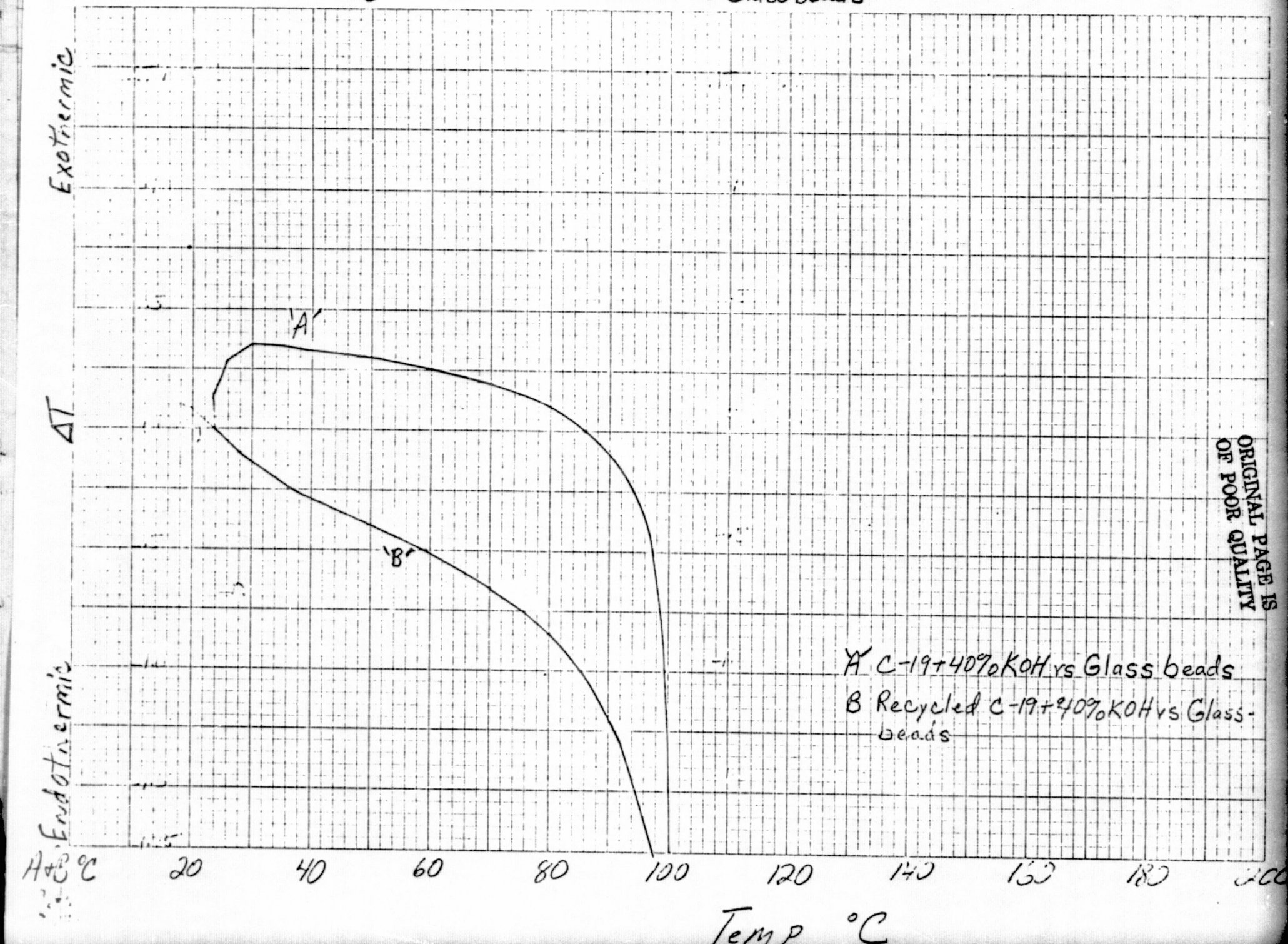
10b C-19 +40% KOH vs Glass beads



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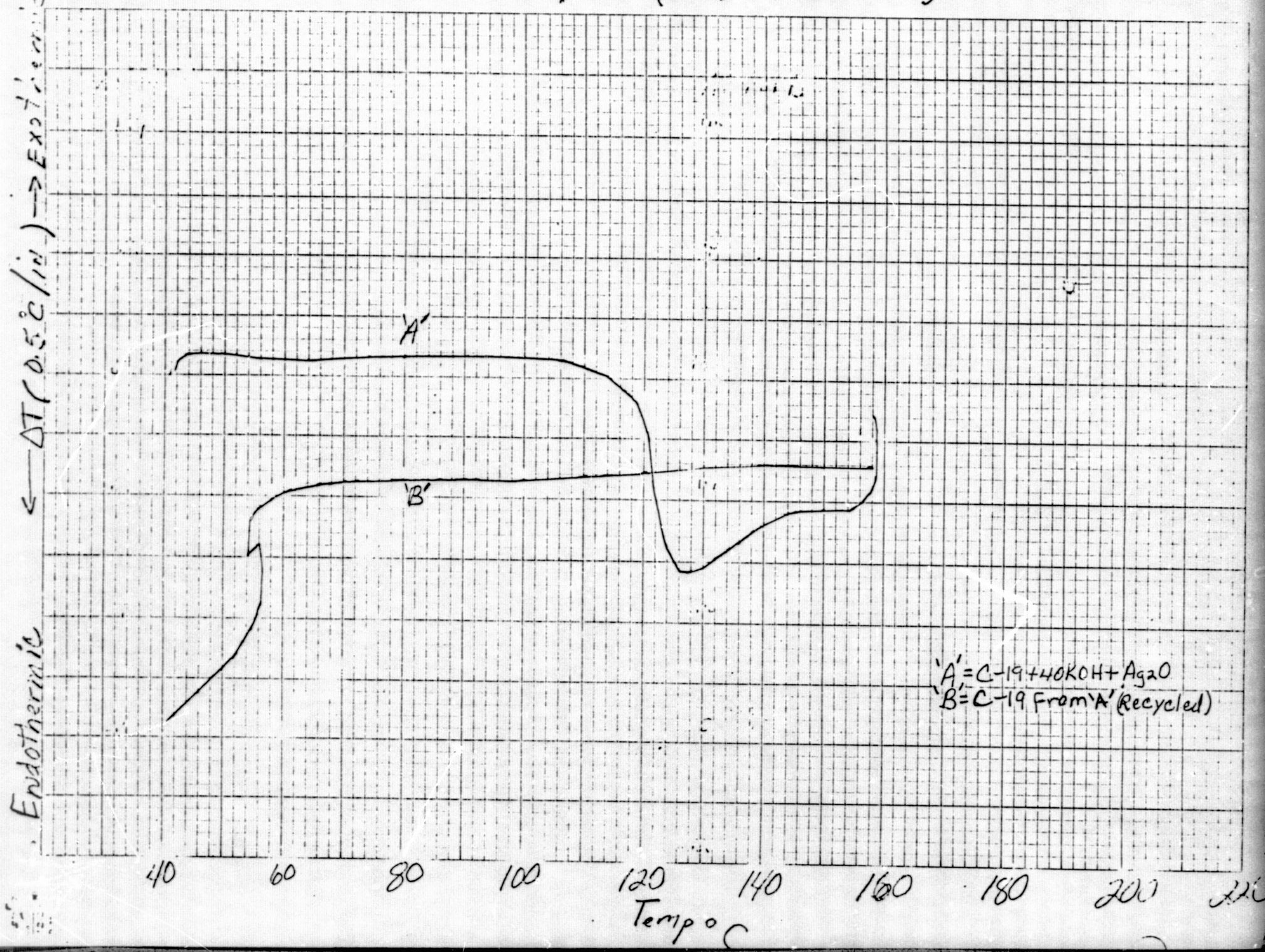
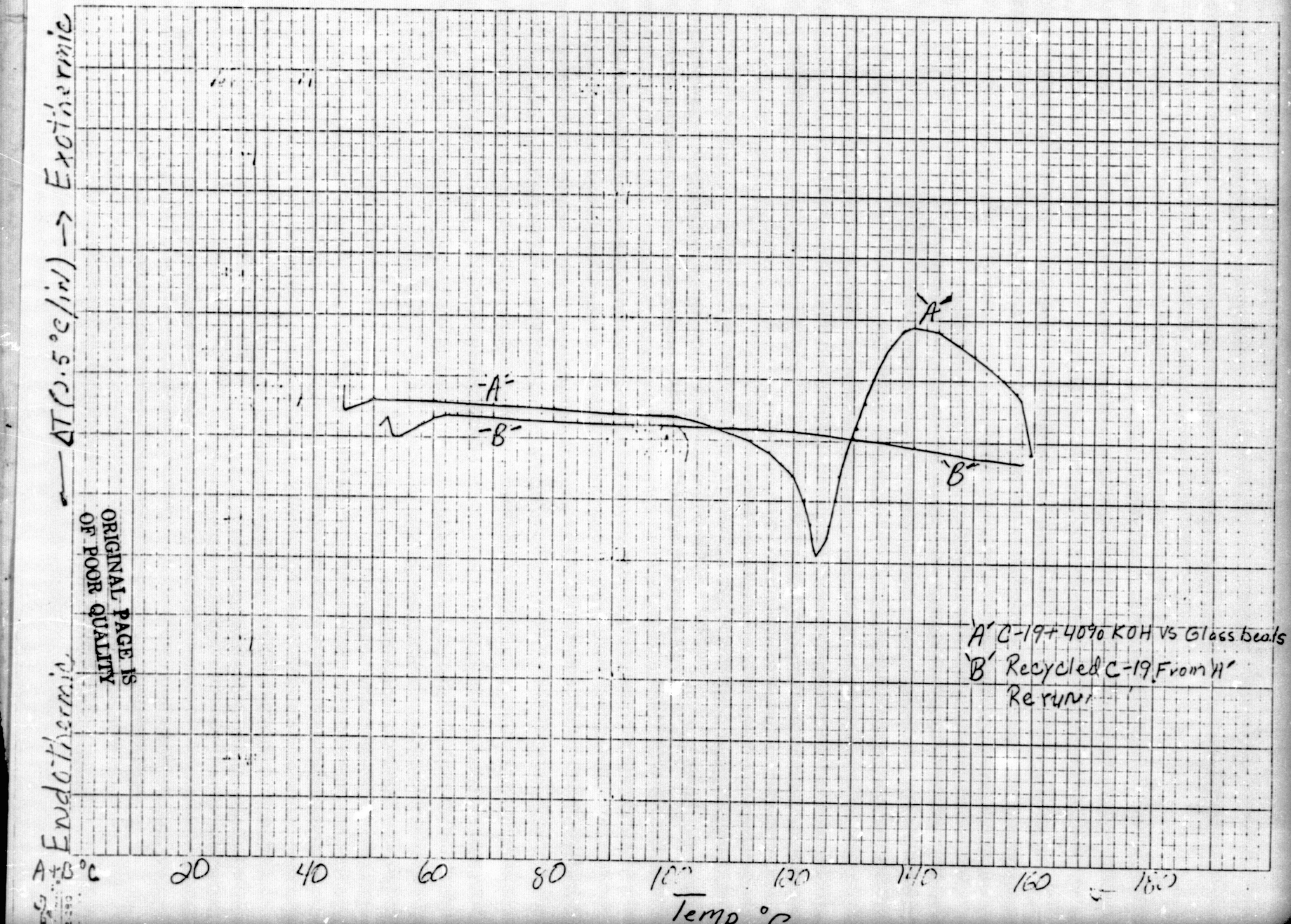
#11 Cellophane (C-19) + 40% KOH + Ag_2O



#12 C-19 +40% KOH vs Glass beads+40% KOH



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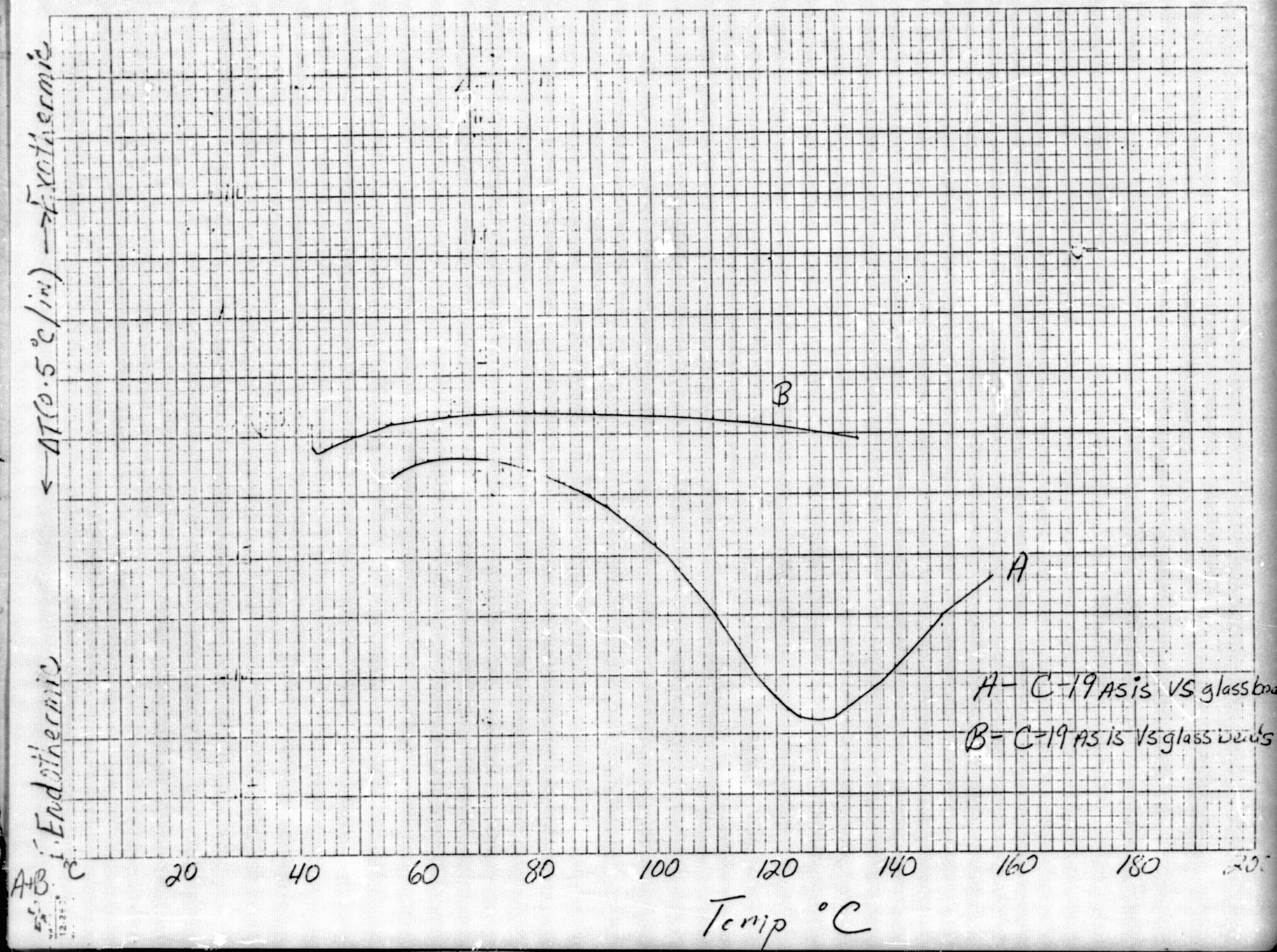
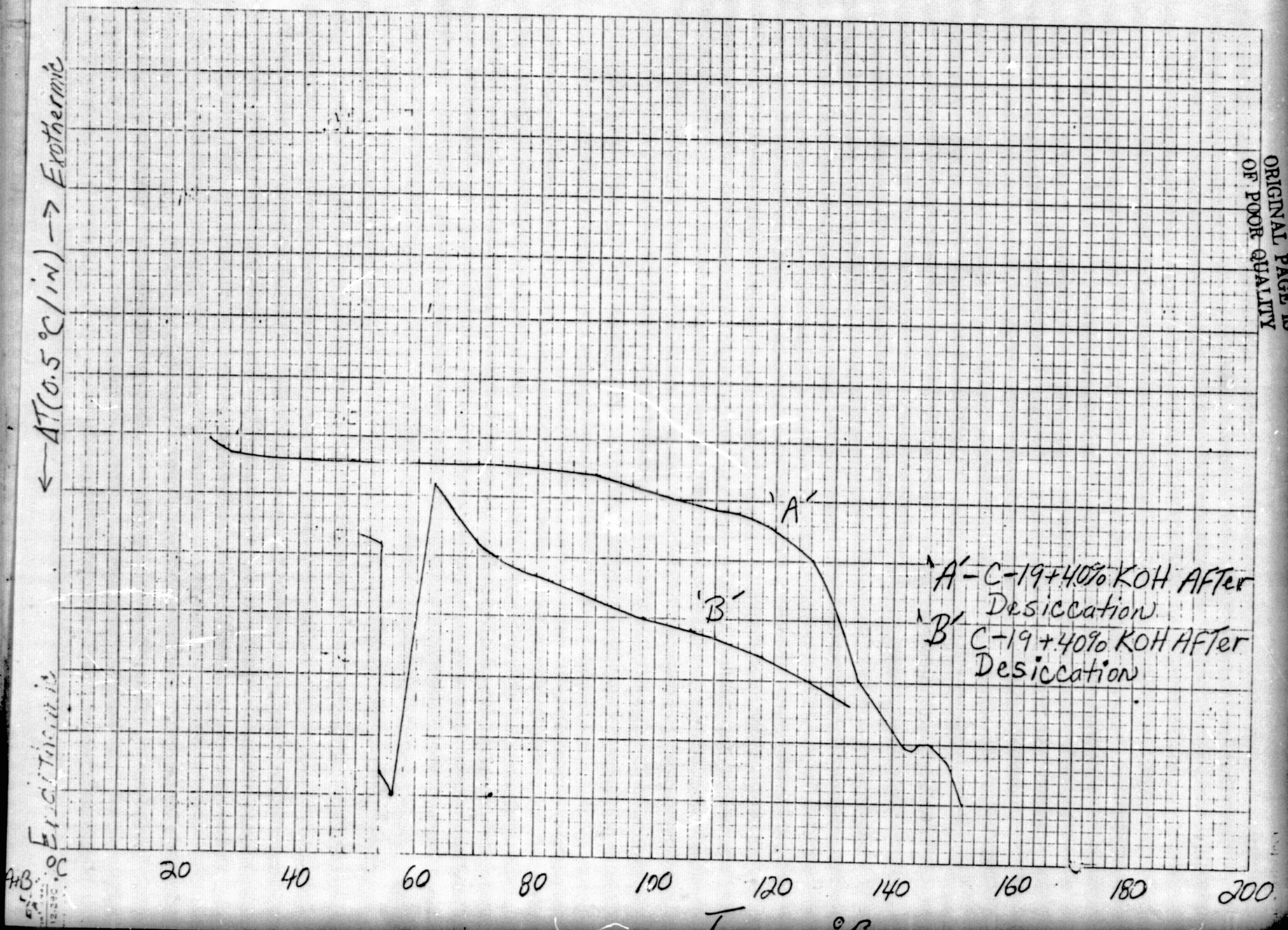
#13


←ΔT(0.5°C/in) →Exothermic
Endothermic
B
A
A- C-19 As is VS glass boa
B- C-19 As is Vs glass beads
20
40
60
80
100
120
140
160
180
Temp °C

14



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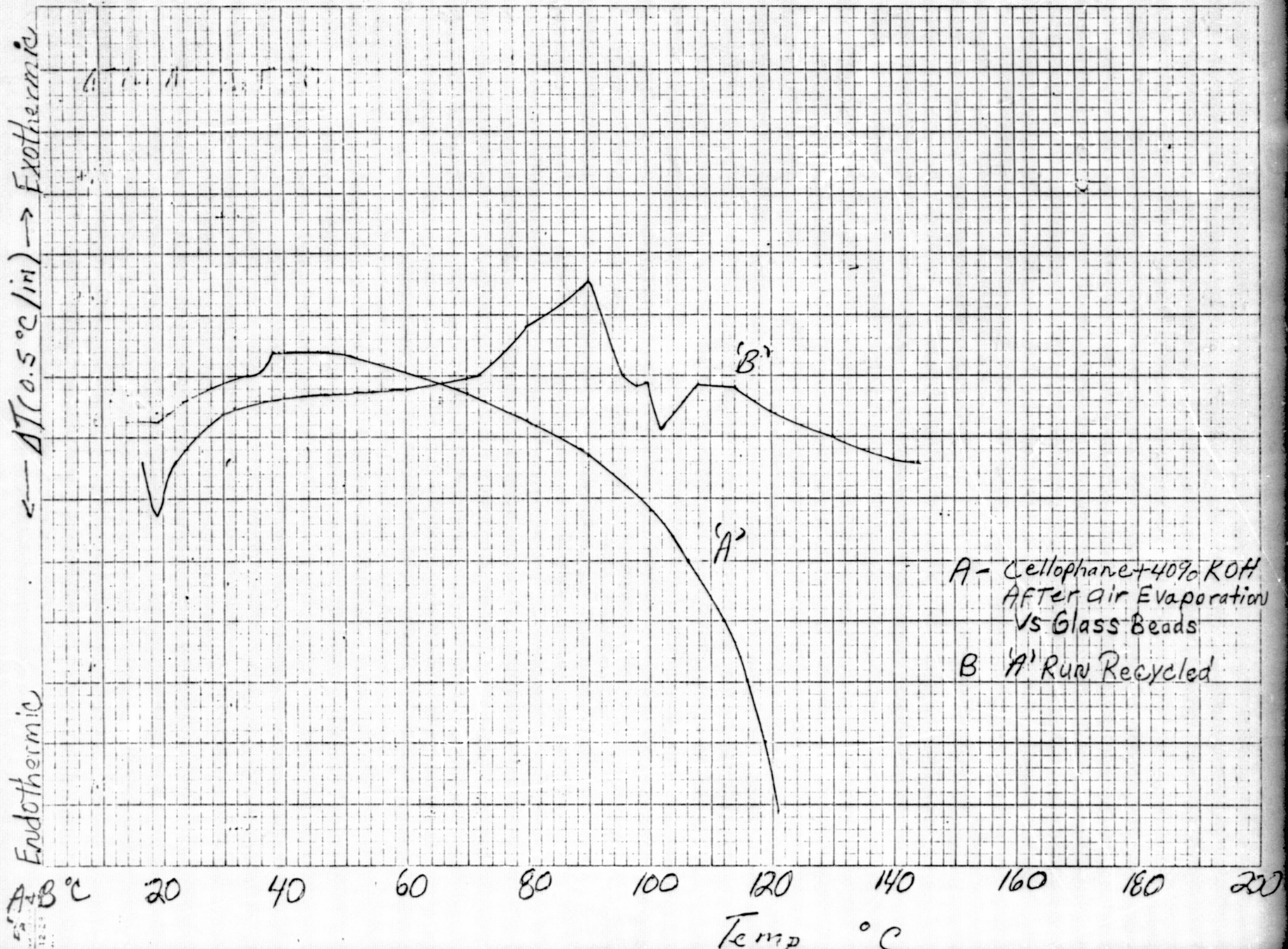
#15


← ΔT (0.5 °C/in) → Exothermic
Endothermic
'B'
'A'
A - Cellophane + 40% KOH
After air Evaporation
Vs Glass Beads
B 'A' Run Recycled
A+B °C
20
40
60
80
100
120
140
160
180
200
Temp °C

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#17

A- C-19 + 40% KOH + Ag2O After Desiccation

B- Recycled C-19 From A - Rerun

A'

B'

B

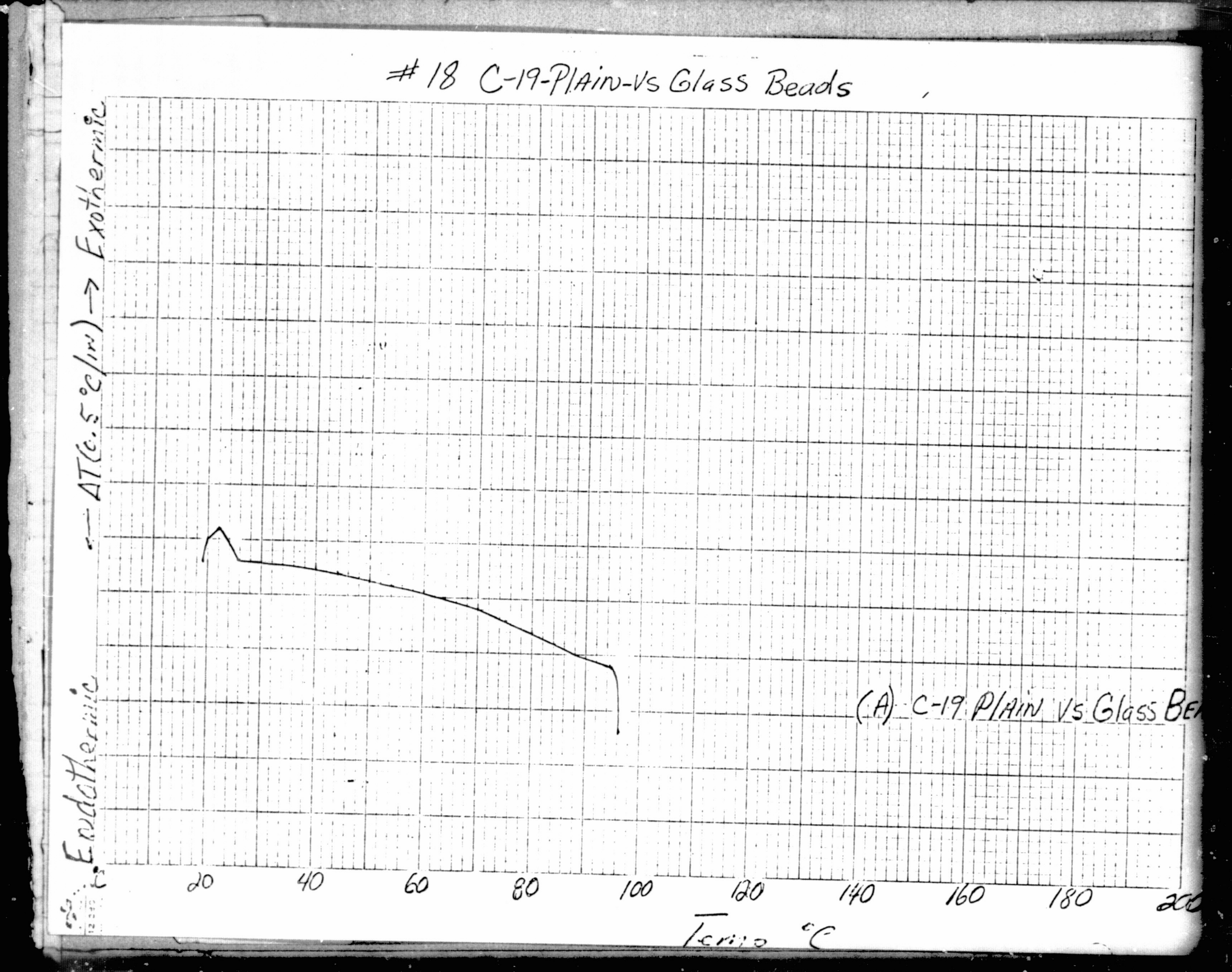
A

20 40 60 80 100 120 140 160 180

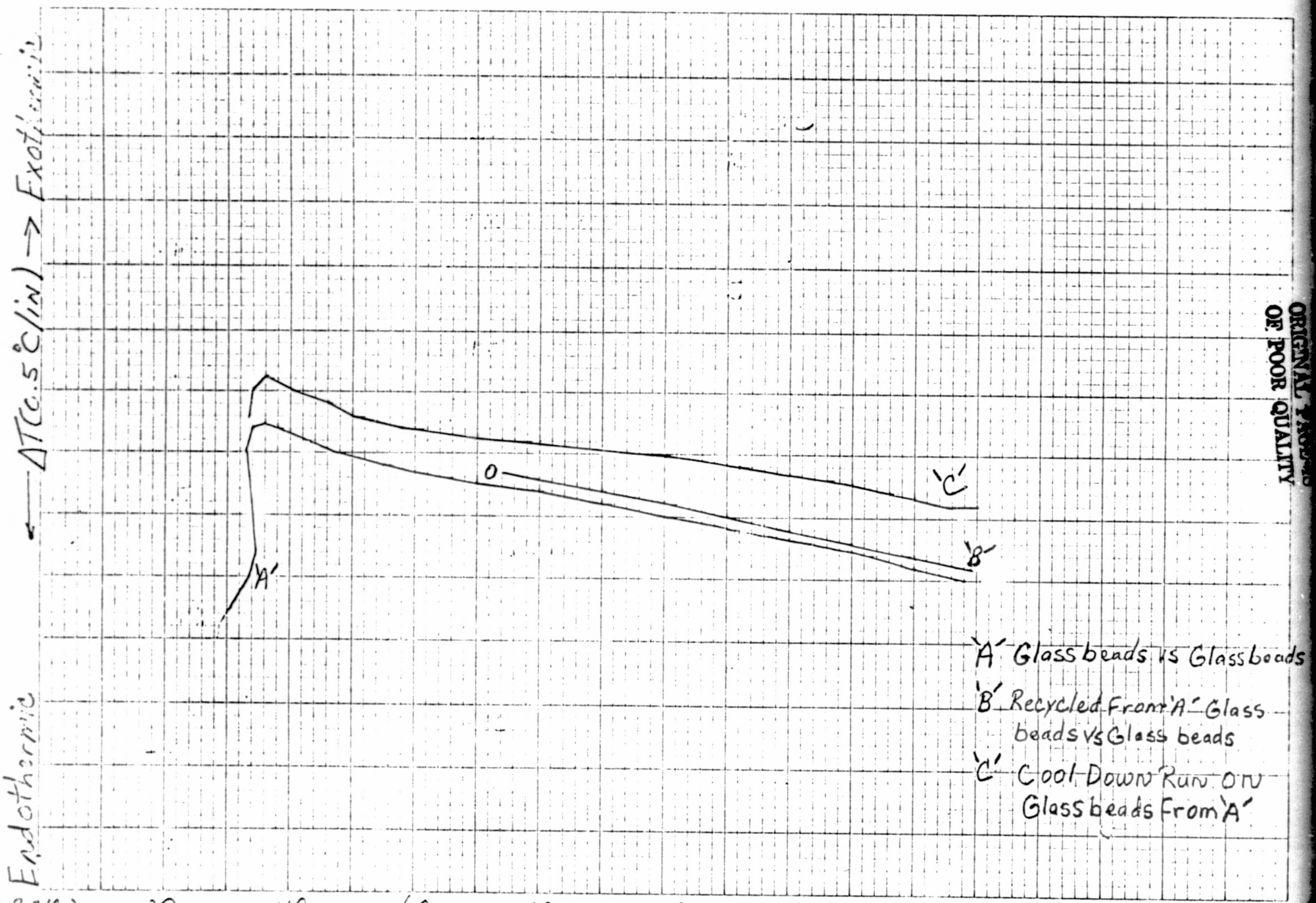
Endothermic

← ΔT (0.5 °C/in) → Exothermic

#18 C-19-Plain-Vs Glass Beads



#19 Glass beads vs Glass beads



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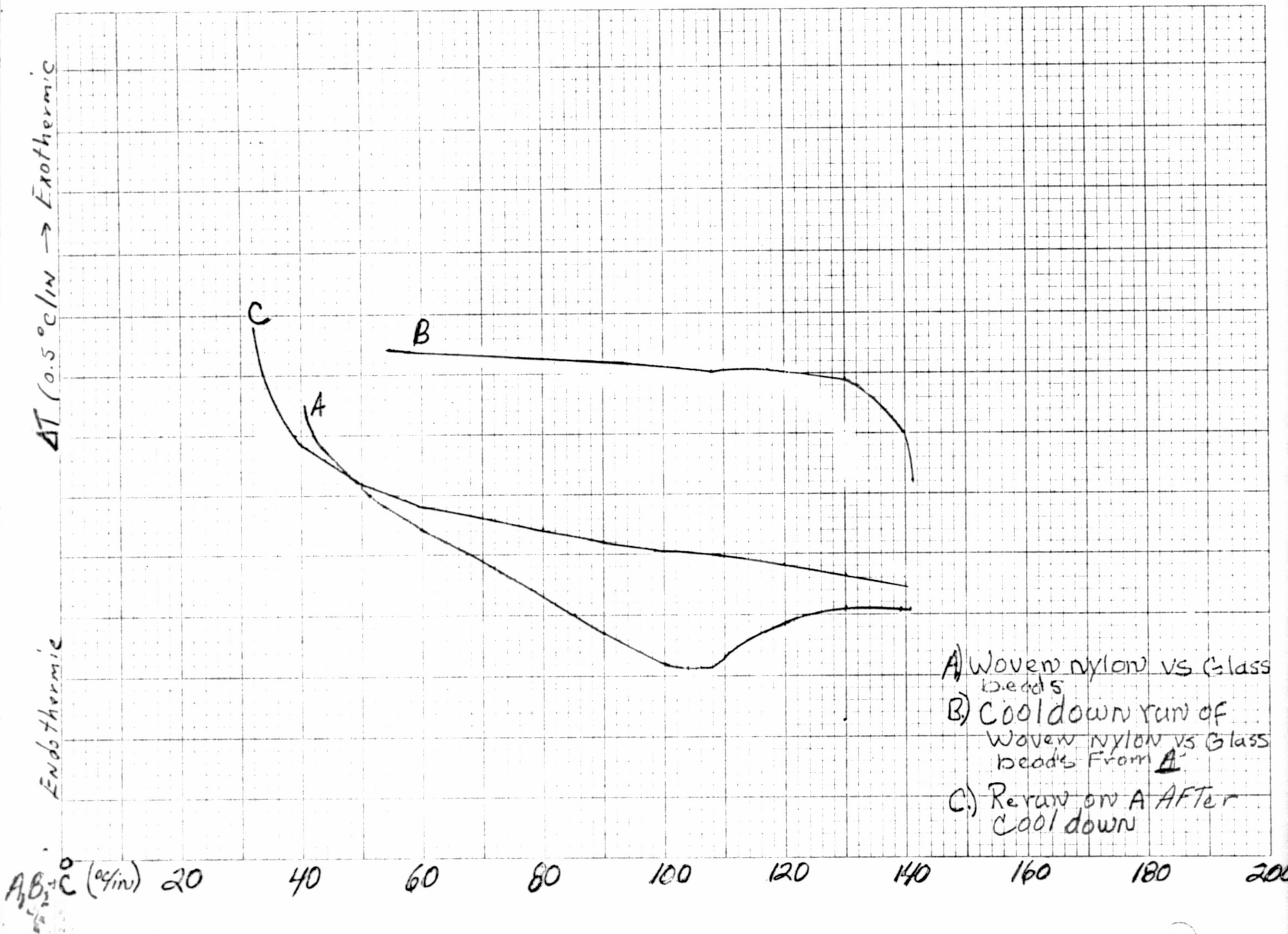
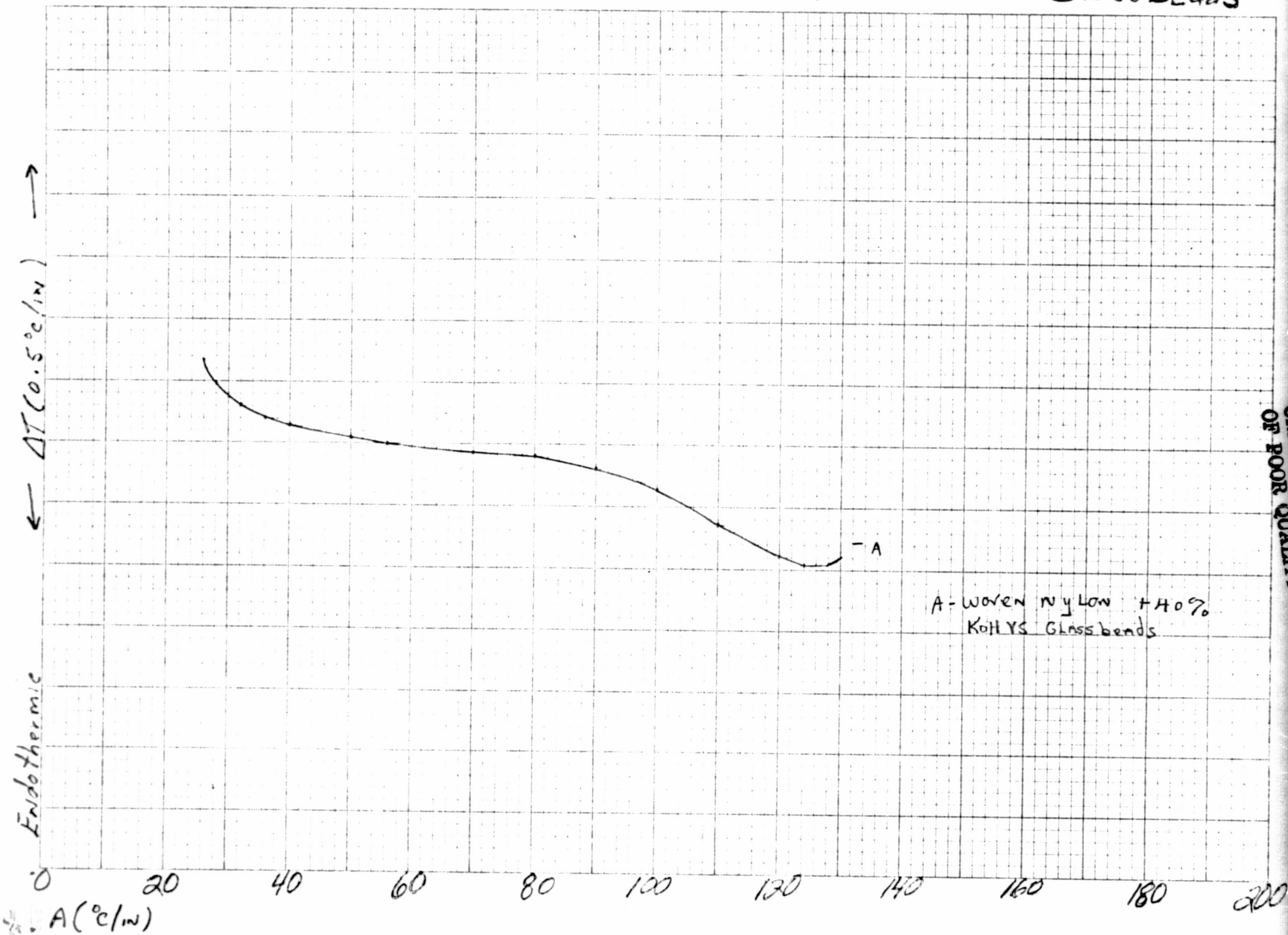
#20


ΔT (0.5 °C/in → Exothermic
Endothermic
A, B, C
°C (°C/in)
20
40
60
80
100
120
140
160
180
200
C
B
A
A) Woven nylon vs Glass beads
B) Cooldown run of Woven Nylon vs Glass beads From A
C) Rerun on A After Cool down

#21 Woven nylon + 40% KOH vs Glass beads



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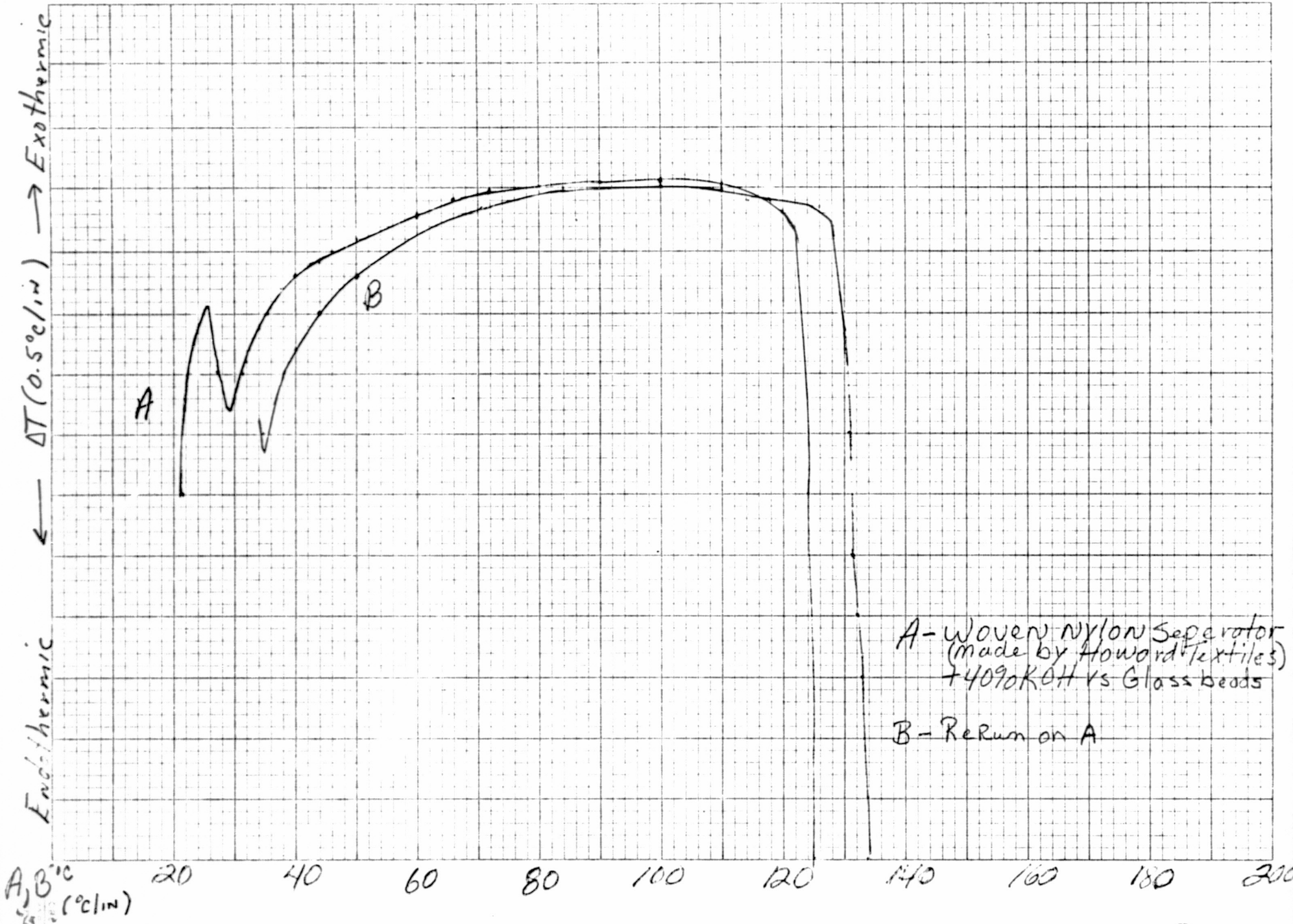
#22 Woven nylon separator



10 Squares to the Inch

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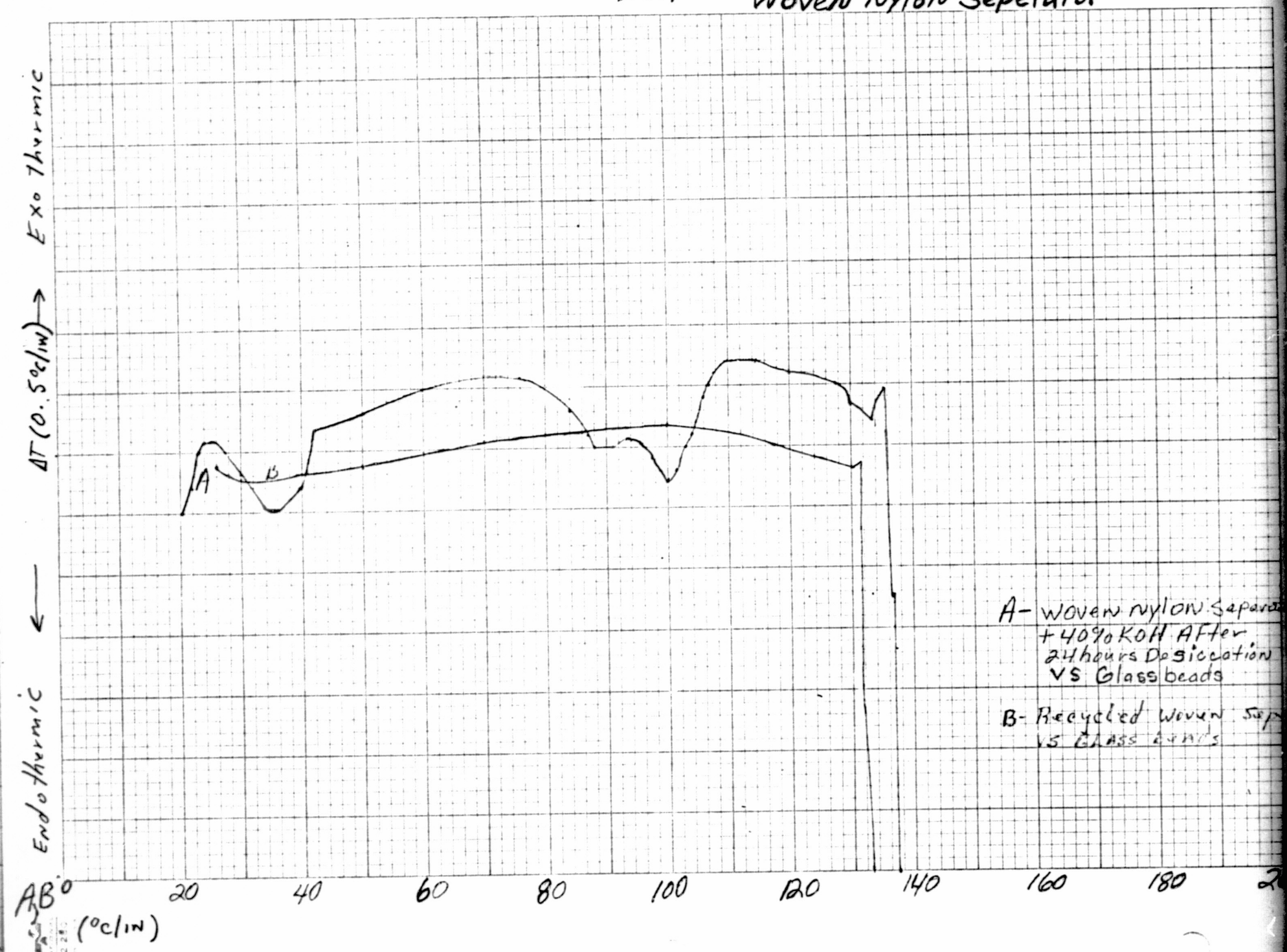
#23 Woven nylon Seperator

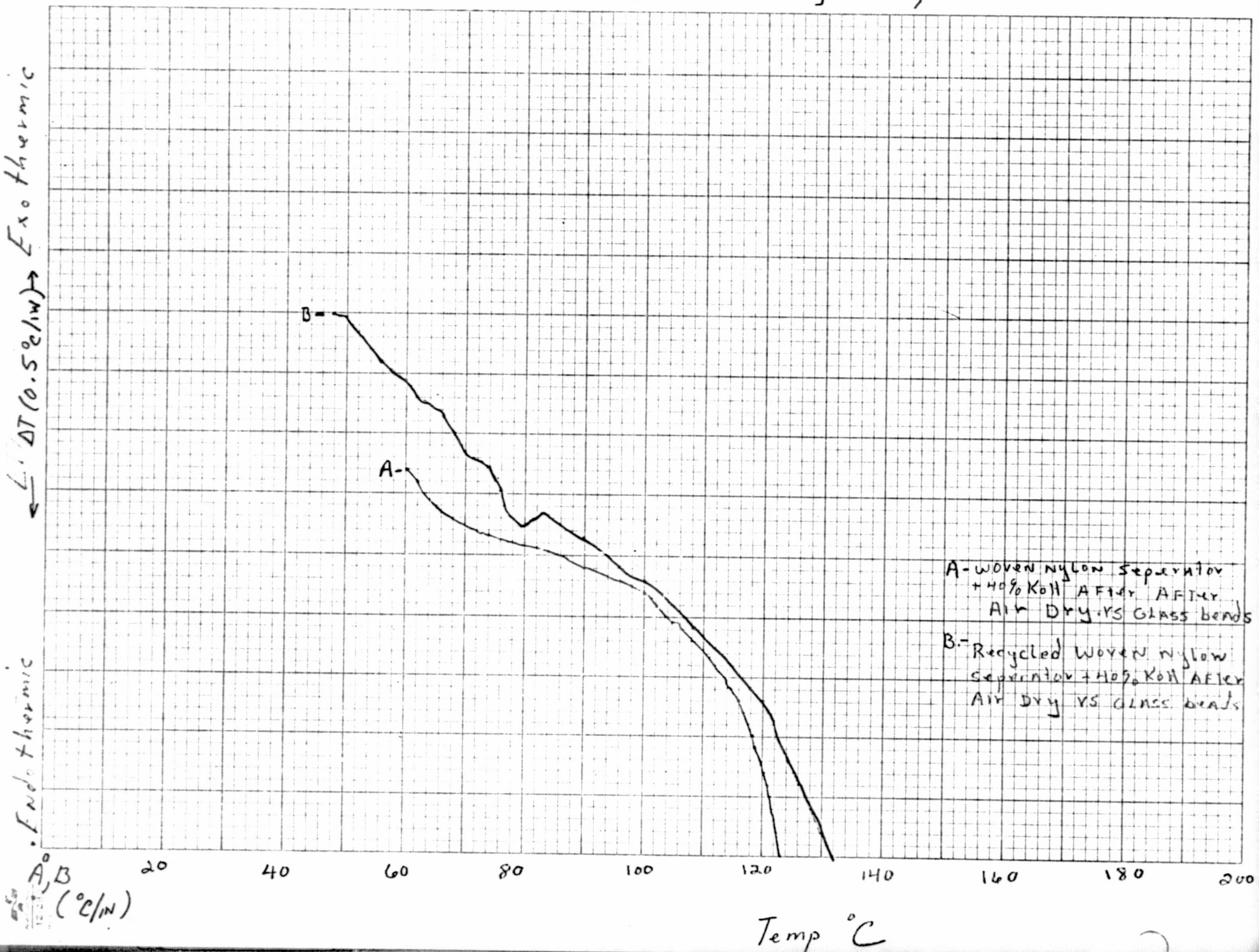
A-Woven nylon Seperator(as is) VS Glass beads

B-Rerun on A

#24 Woven nylon Seperator



#25 Woven Nylon Seperator



#26 Woven Nylon Separator After 24 hr. Desiccation

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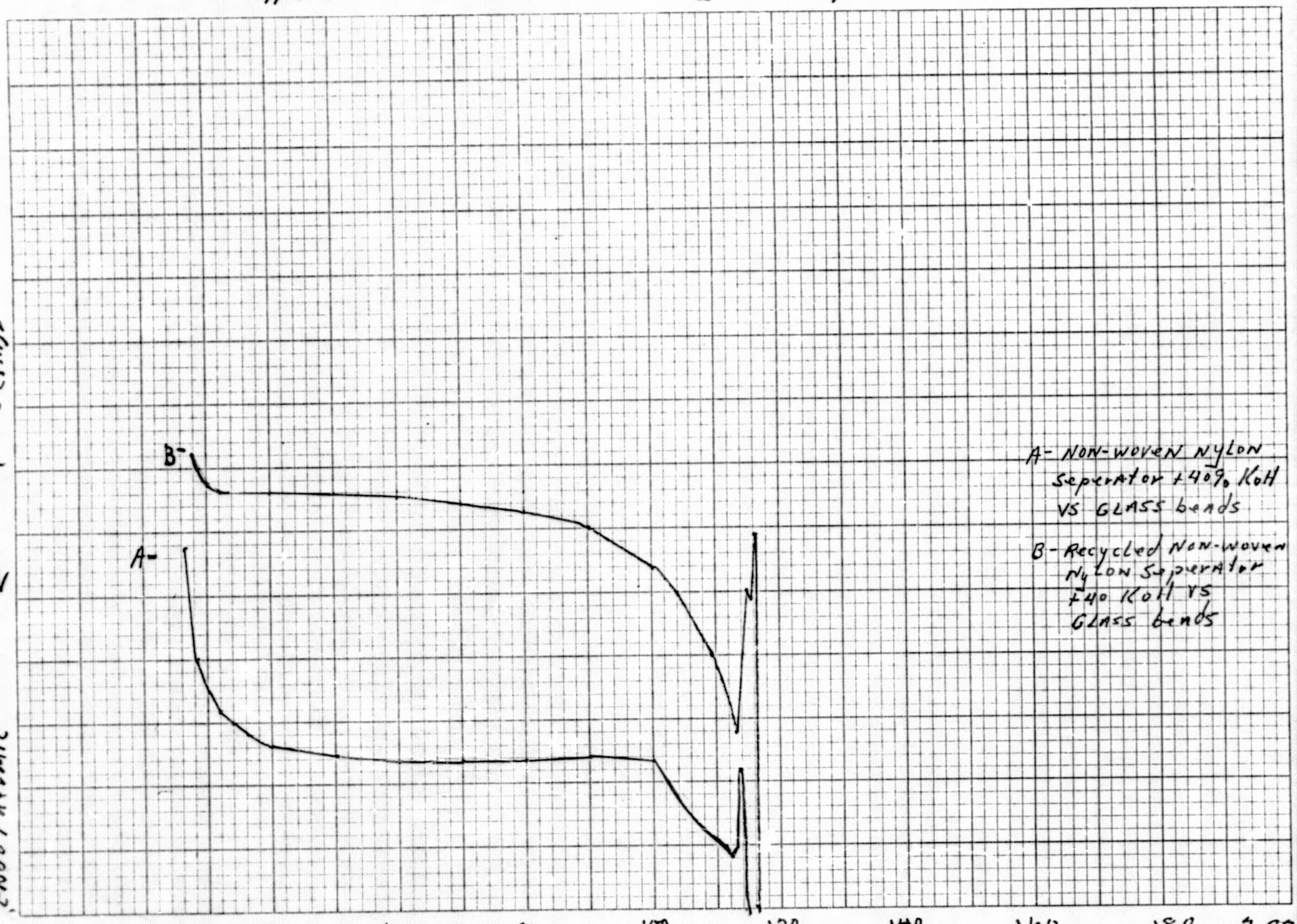
Squares to the Inch

#27 NON-WOVEN NYLON SEPERATOR +KOH 40%



#28 NON-WOVEN NYLON SEPERATOR PLAIN

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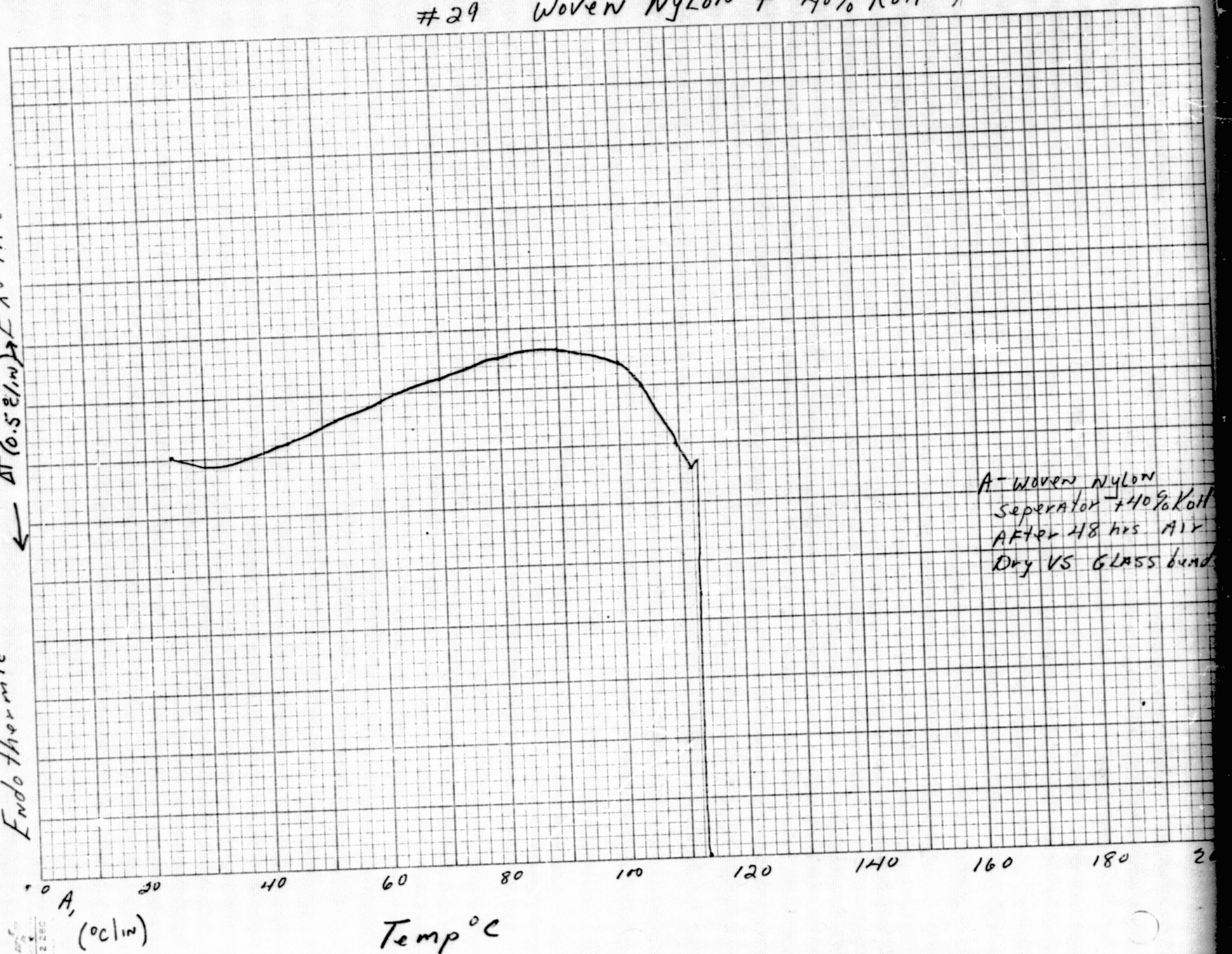
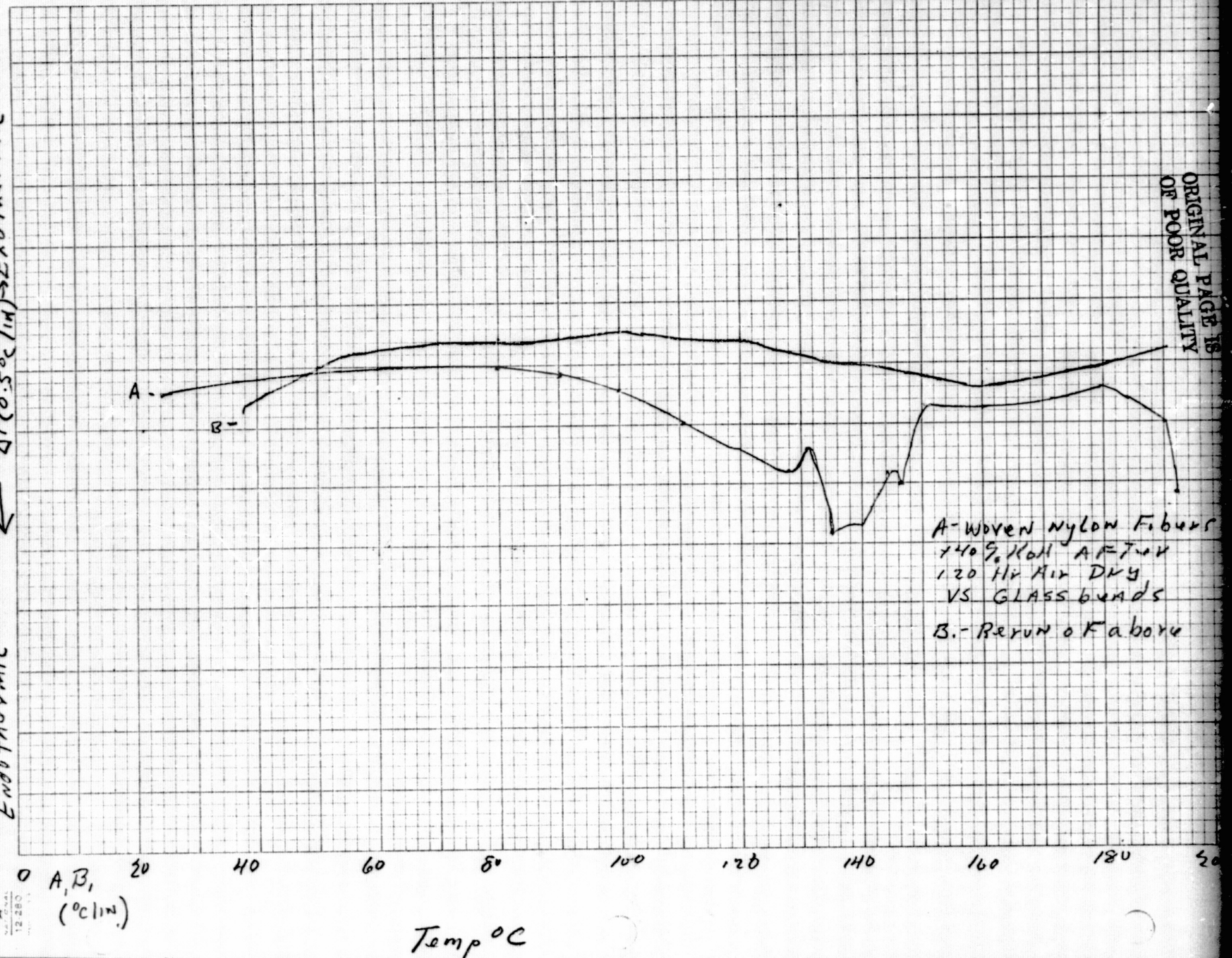
#29 Woven Nylon + 40% KOH



#131 Woven Fibers +40% Roll AFTer 120 hr Air



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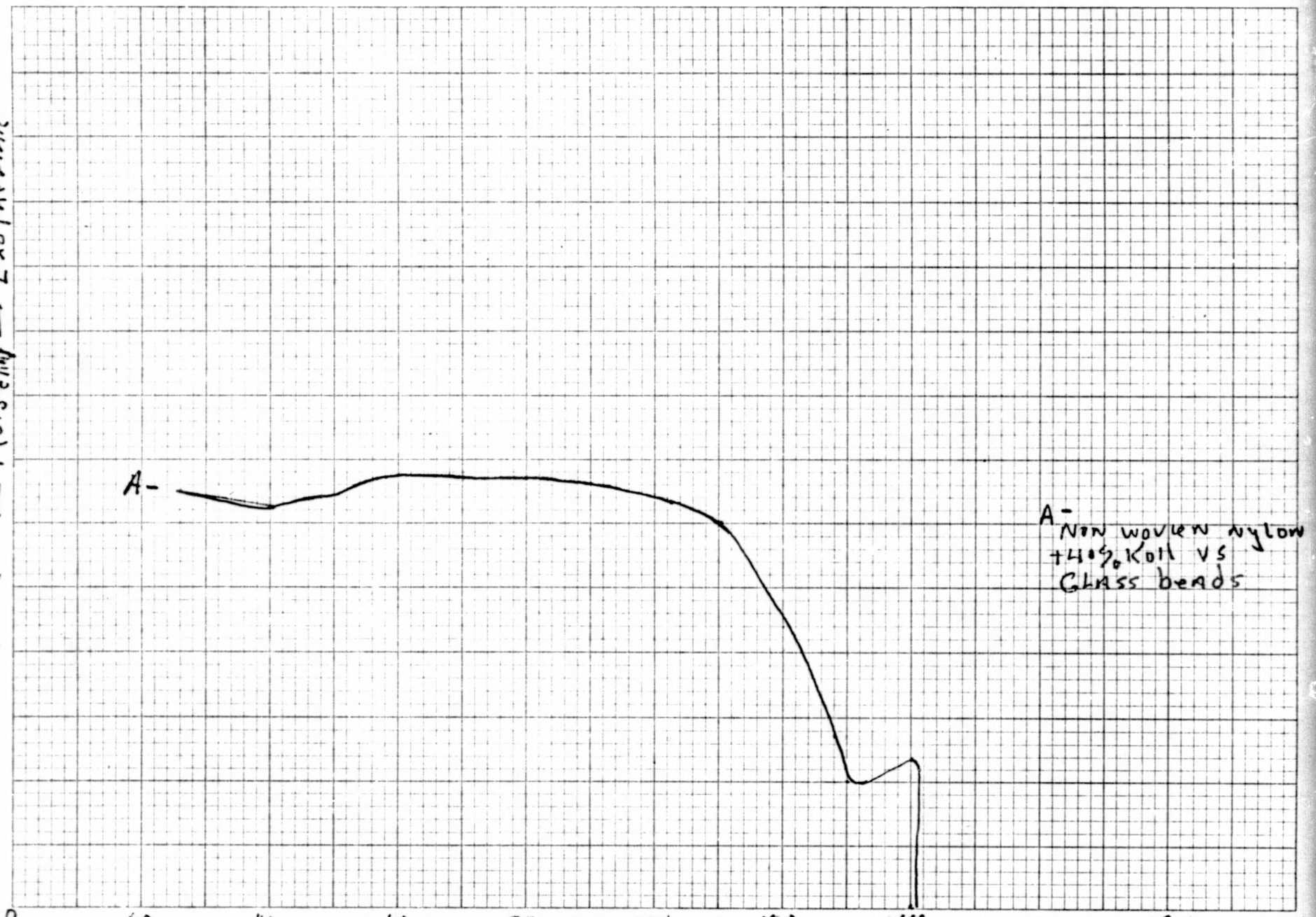
NON-WOVEN NYLON #33



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#34

1018 Tape

A- 1018 Tape vs Glass beads

B- Rerun of above

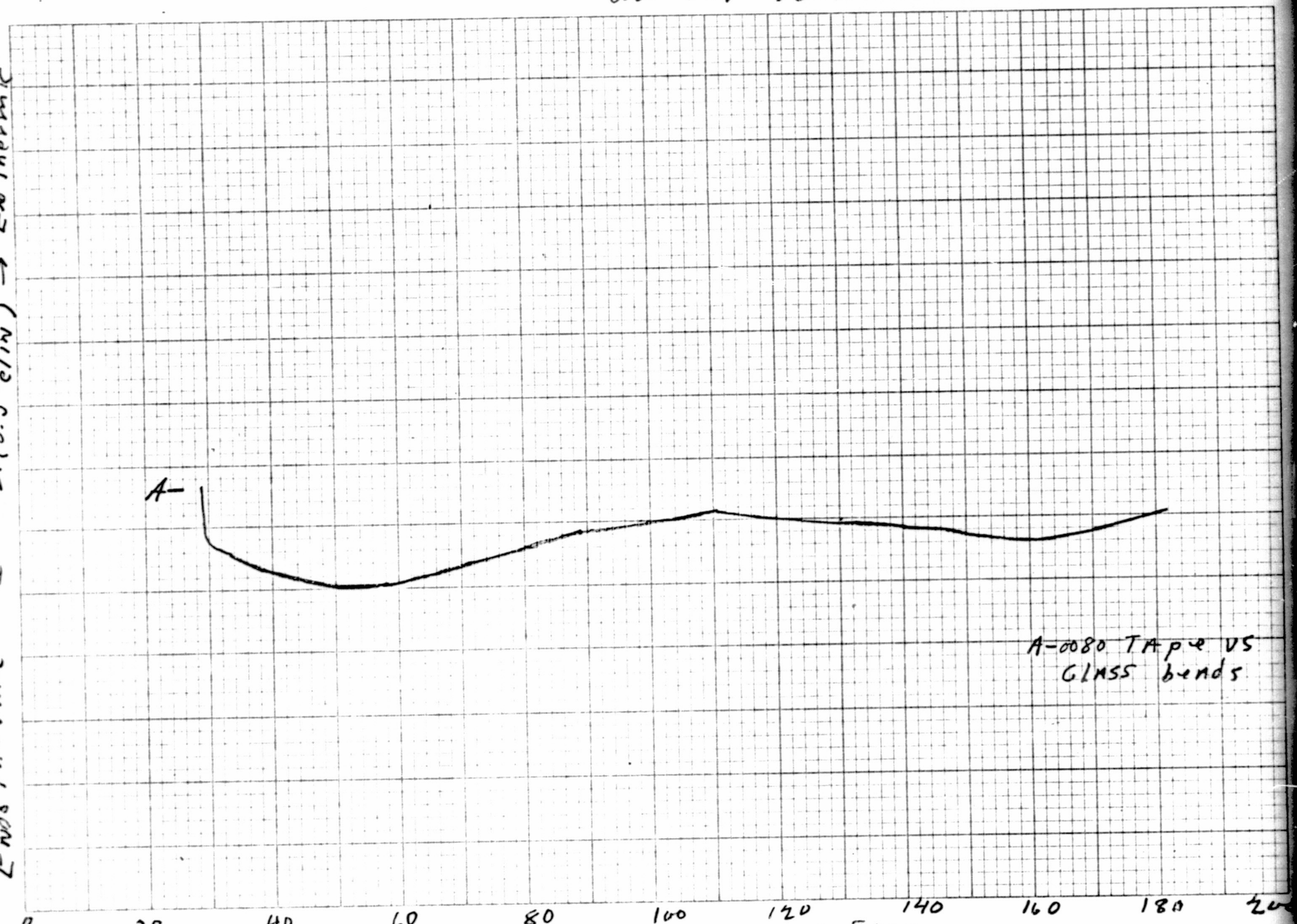
1018 Tape from Vitta Corp.

Endothermic ← ΔT (0.5°C/in) → Exothermic

A, B (10°C/in)

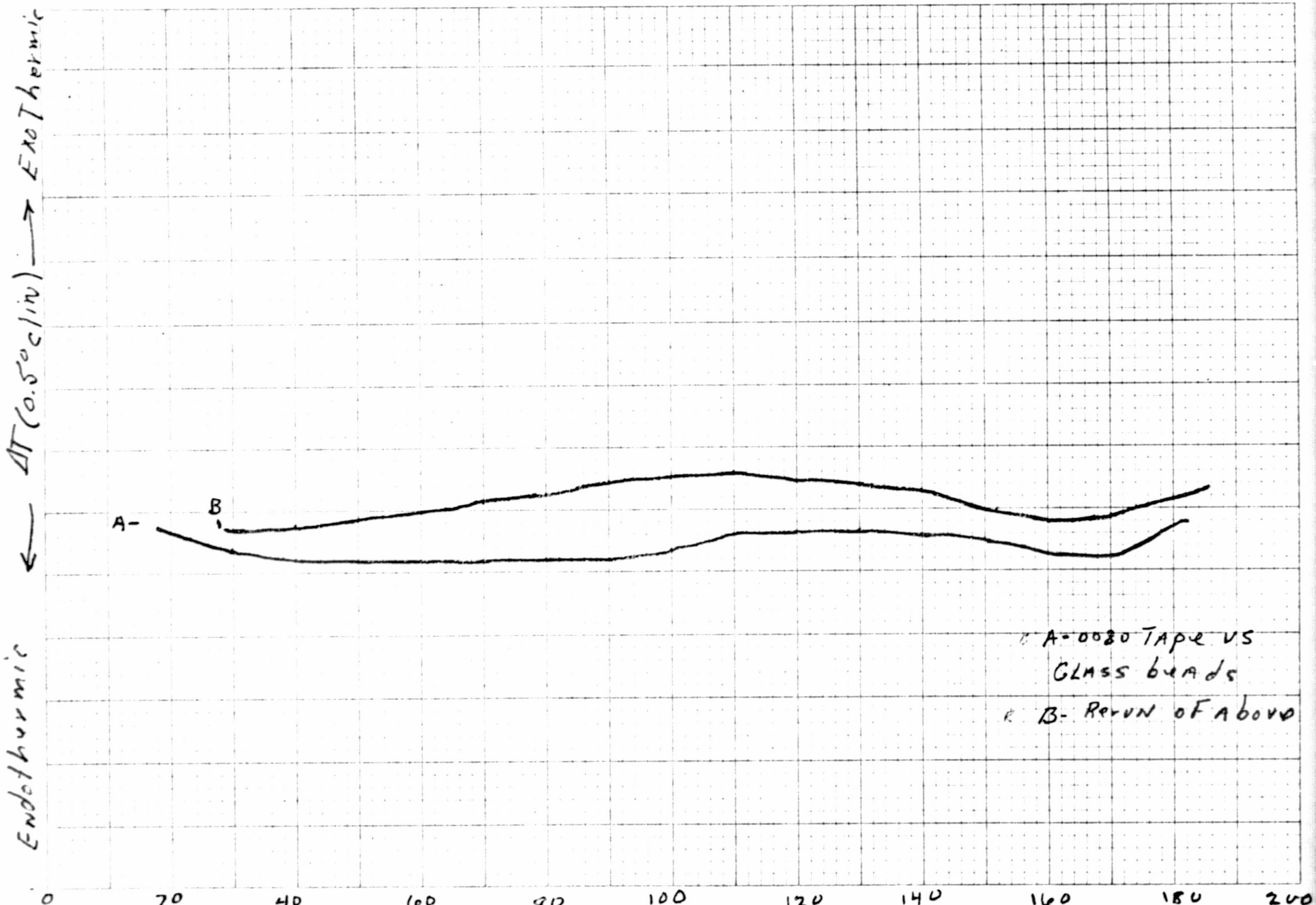
0 20 40 60 80 100 120 140 160 180 200

0080 TAPE VS GLASS bonds #35



RK

0080 TAPE #36



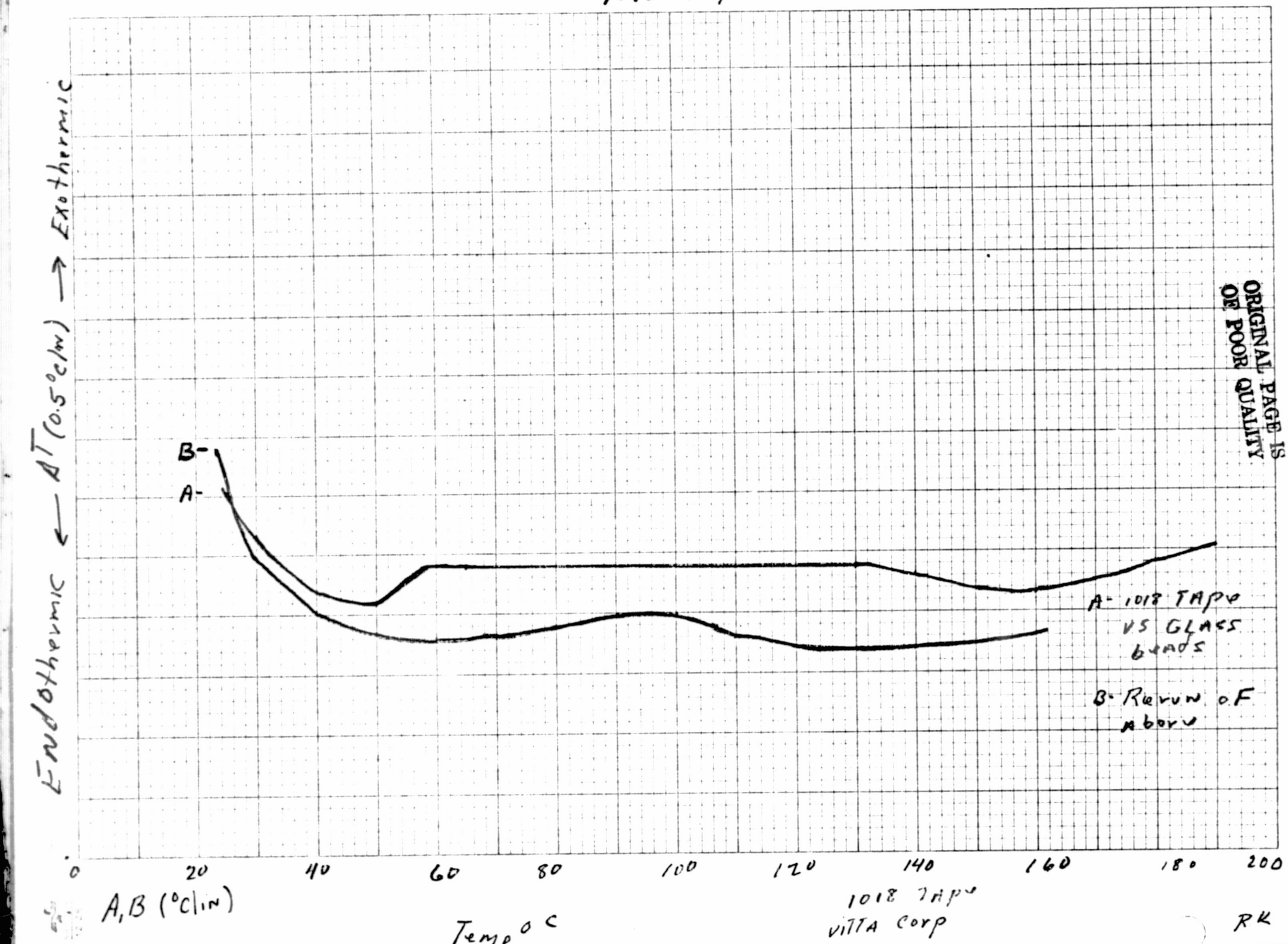
A- 0080 TAPE VS GLASS BEADS

B- RERUN OF ABOVE

R.K.

1018 TAPE

A 38



1018 TAPE
VITTA CORP

RK

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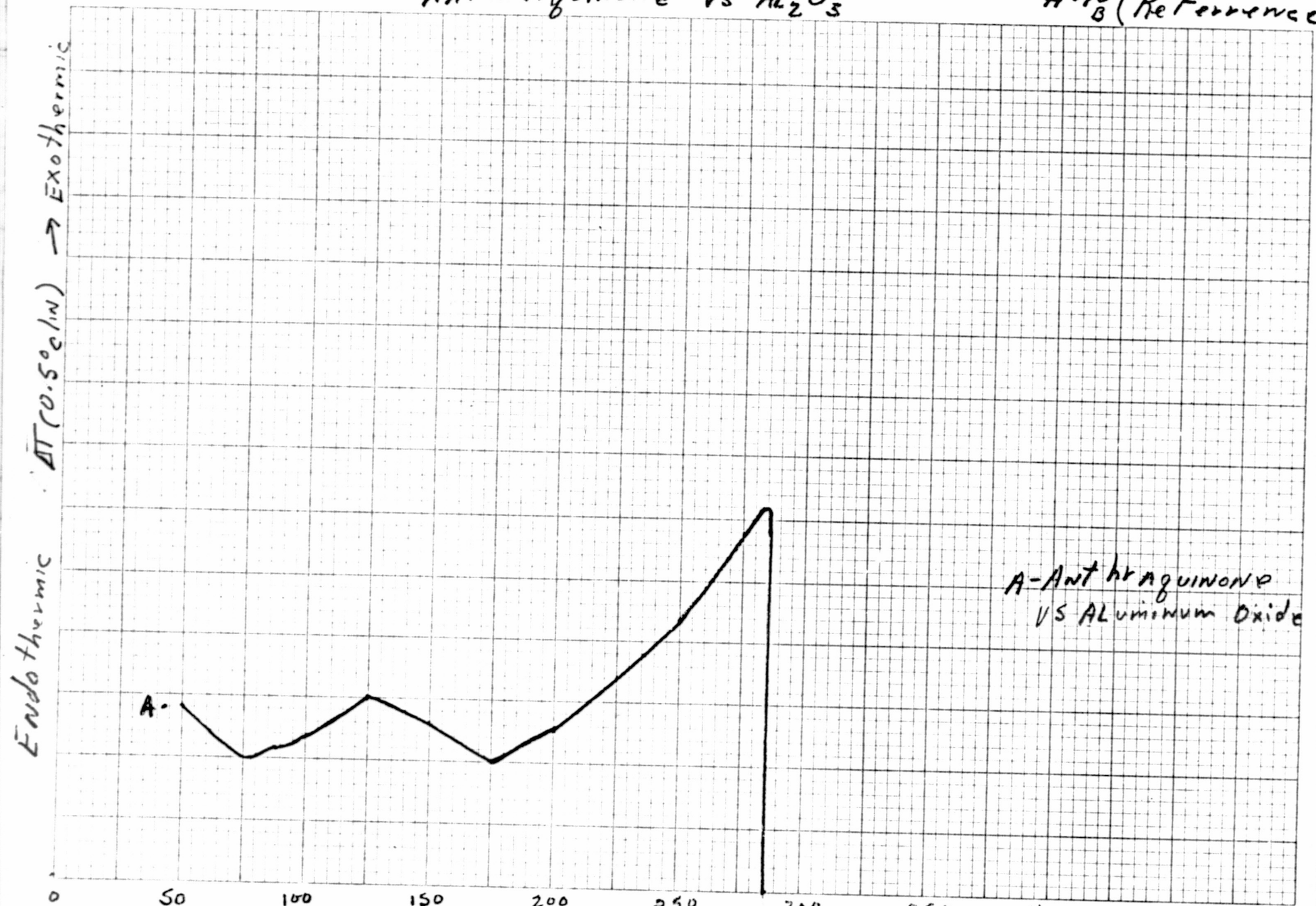
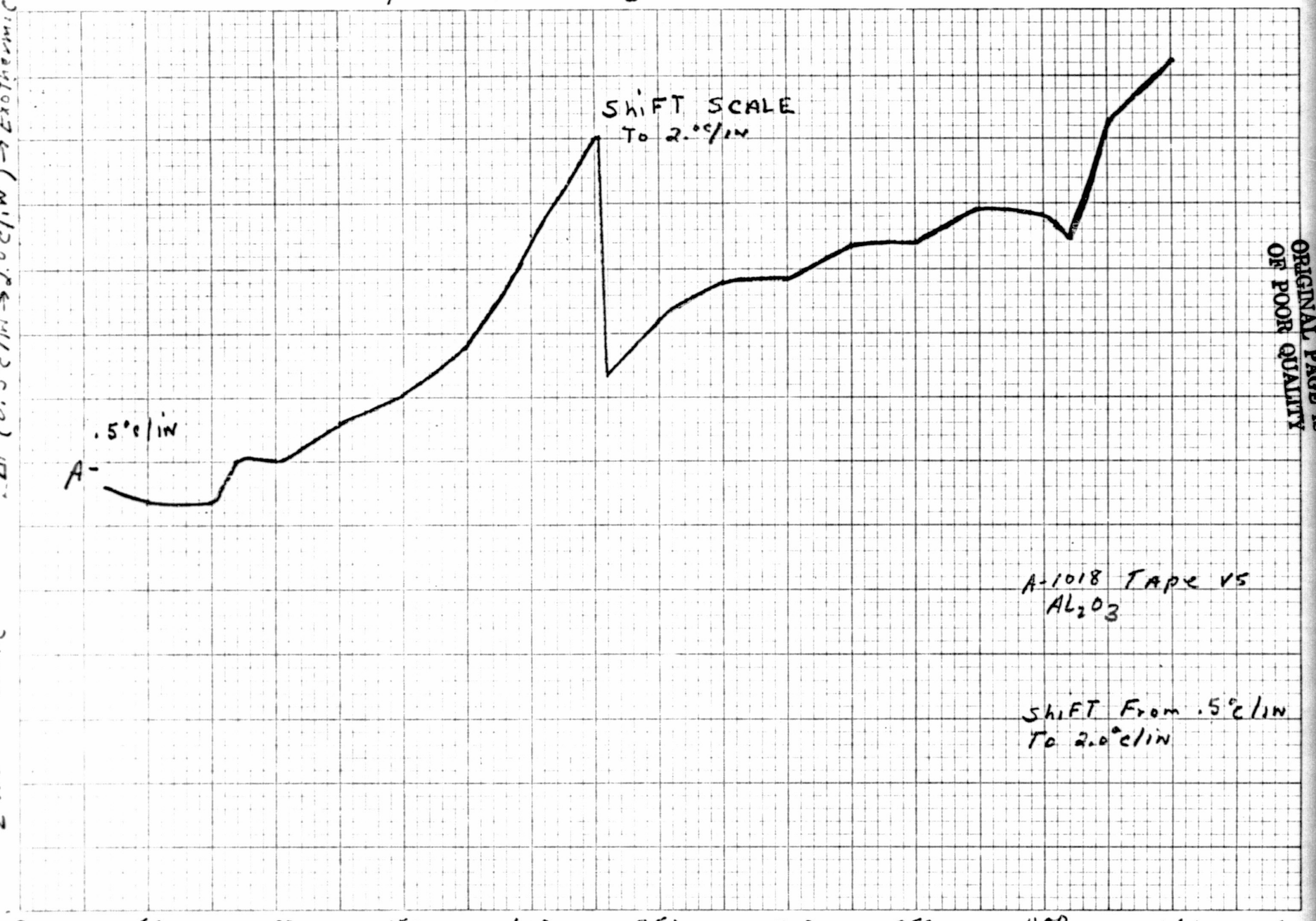
10 Squares to the Inch

Anthraquinone Vs Al_2O_3 #H^0_B (Reference)



1018 Tape Vs Al_2O_3 #41



A-1018 Tape Vs Al_2O_3

Shift From .5°c/in
To 2.0°c/in

1018 Tape

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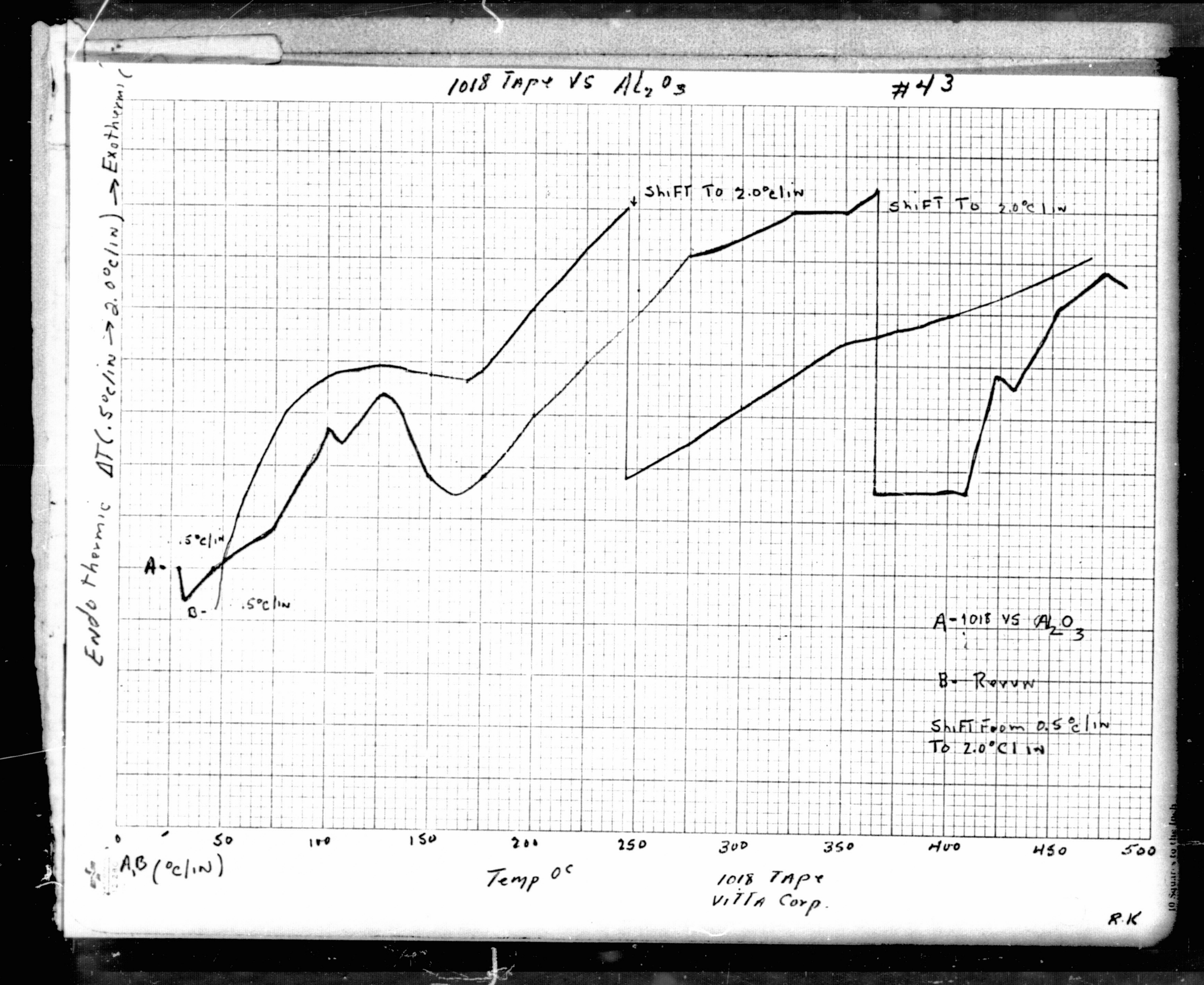
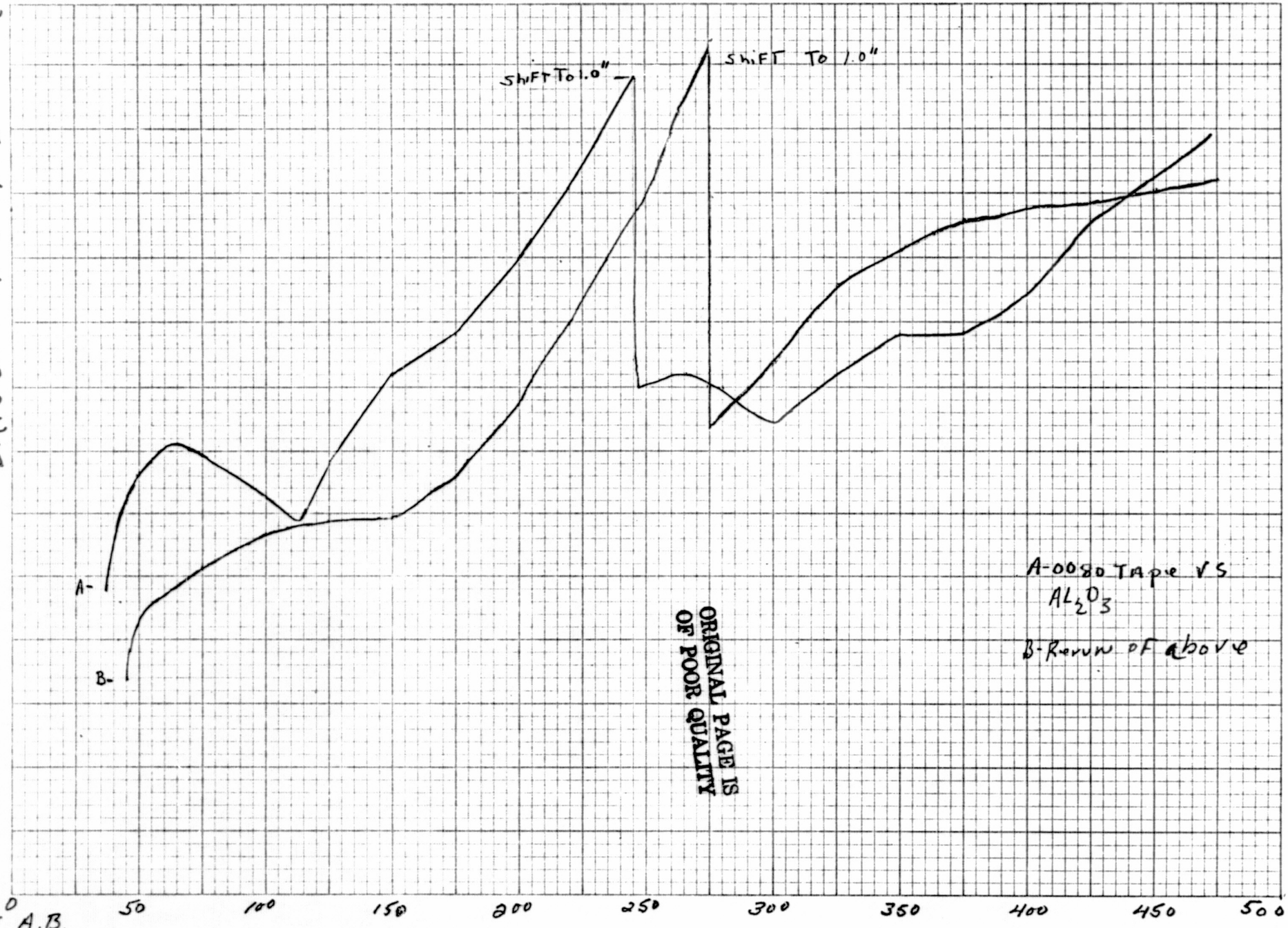

1018 TAPE VS Al_2O_3
#43
Endothermic ΔT(.5°c/in → 2.0°c/in) → Exothermic
SHIFT TO 2.0°c/in
SHIFT TO 2.0°c/in
A-
.5°c/in
B-
.5°c/in
A-1018 VS Al_2O_3
B- Reverse
SHIFT From 0.5°c/in
TO 2.0°c/in
0
50
100
150
200
250
300
350
400
450
500
A,B (°c/in)
Temp °c
1018 TAPE
VITTA Corp.
R.K

0080 Tape Vs AL_2O_3

#44



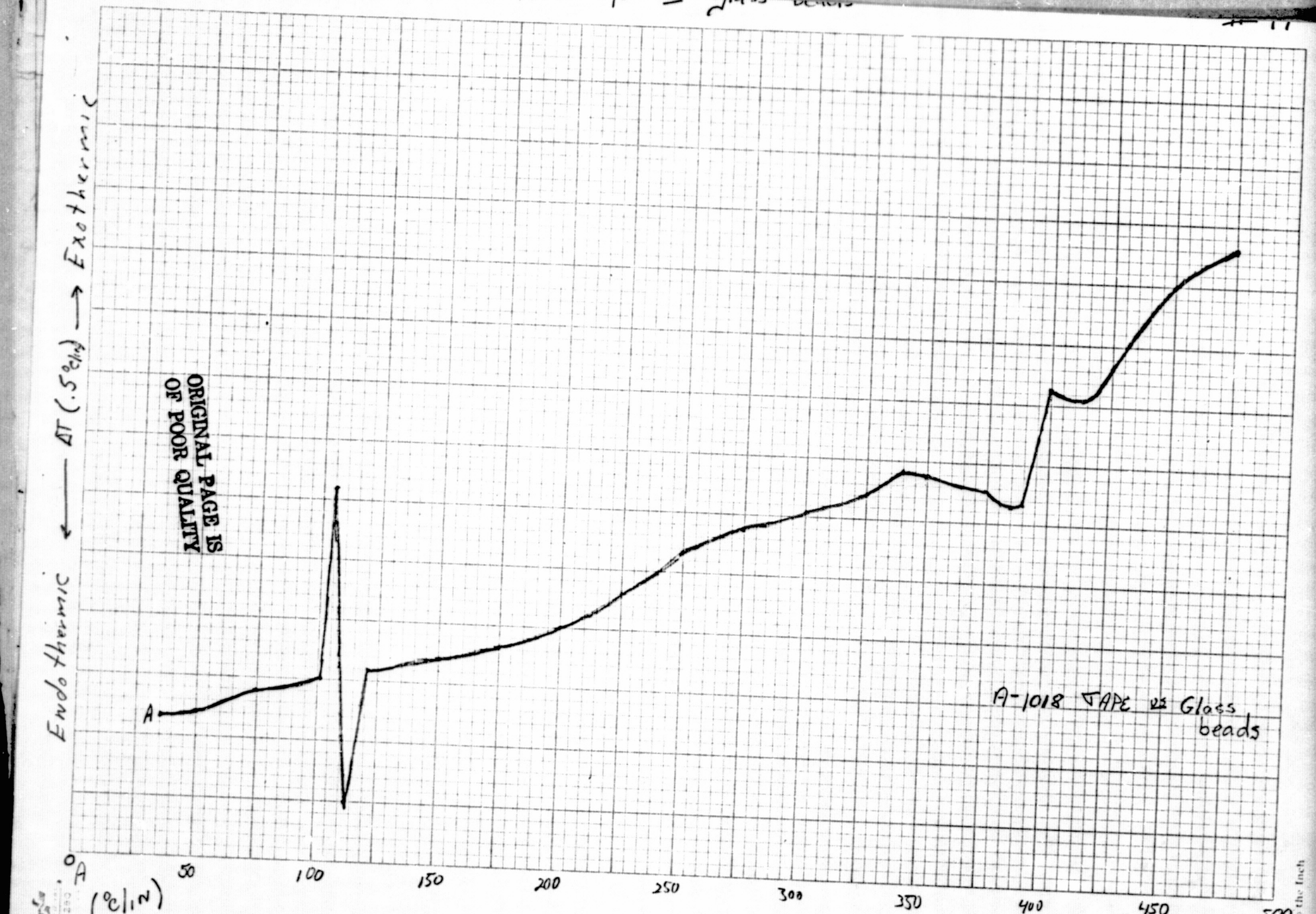
Endothermic ΔT(.5°C/IN → 1.0°C/IN) → Exothermic

0080 Tape
VITTA Corp.

RK.

10 Squares to the Inch

0080 TAPE vs Al_2O_3 #45



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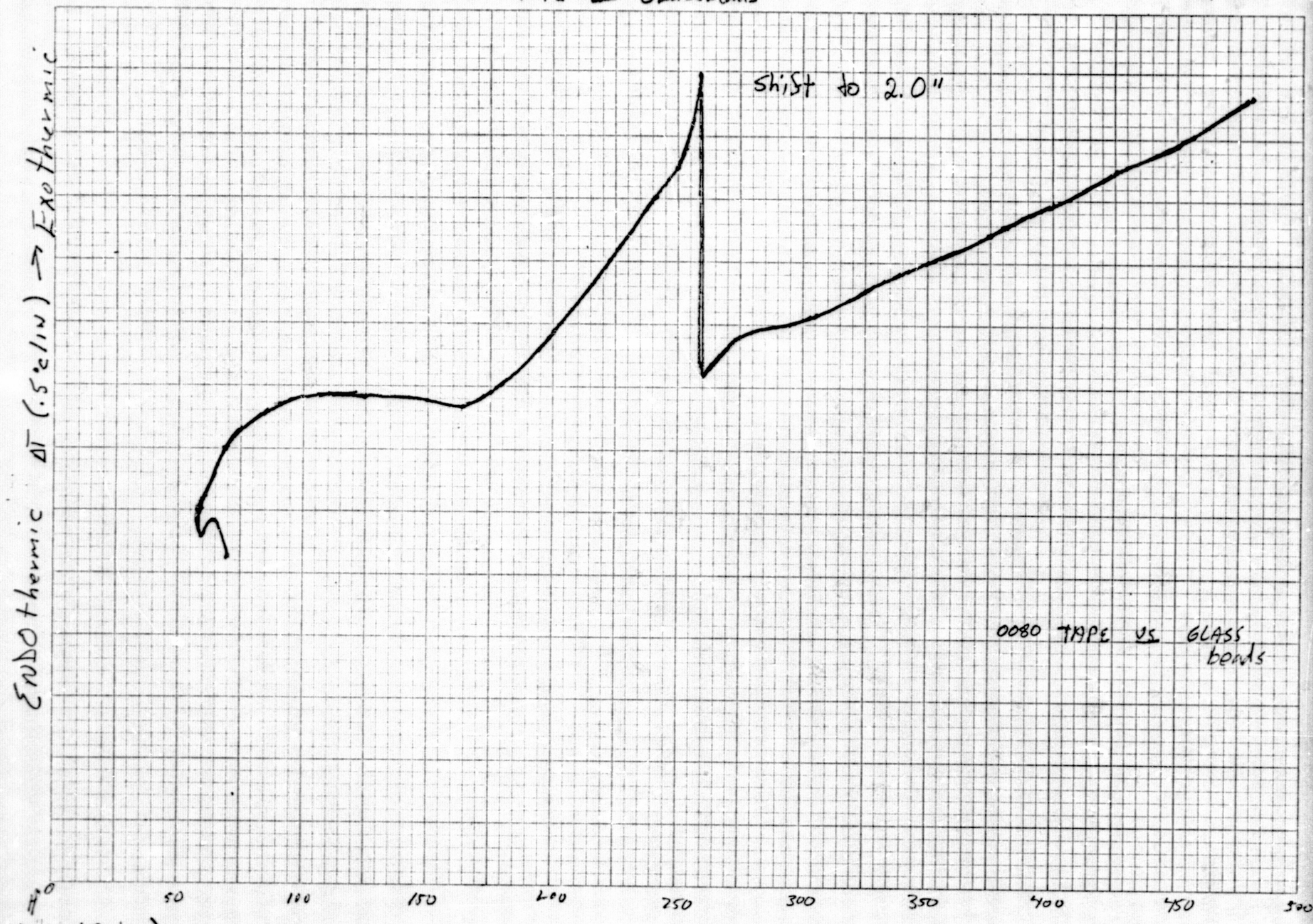
0080 TAPE vs Glassbeads

#48

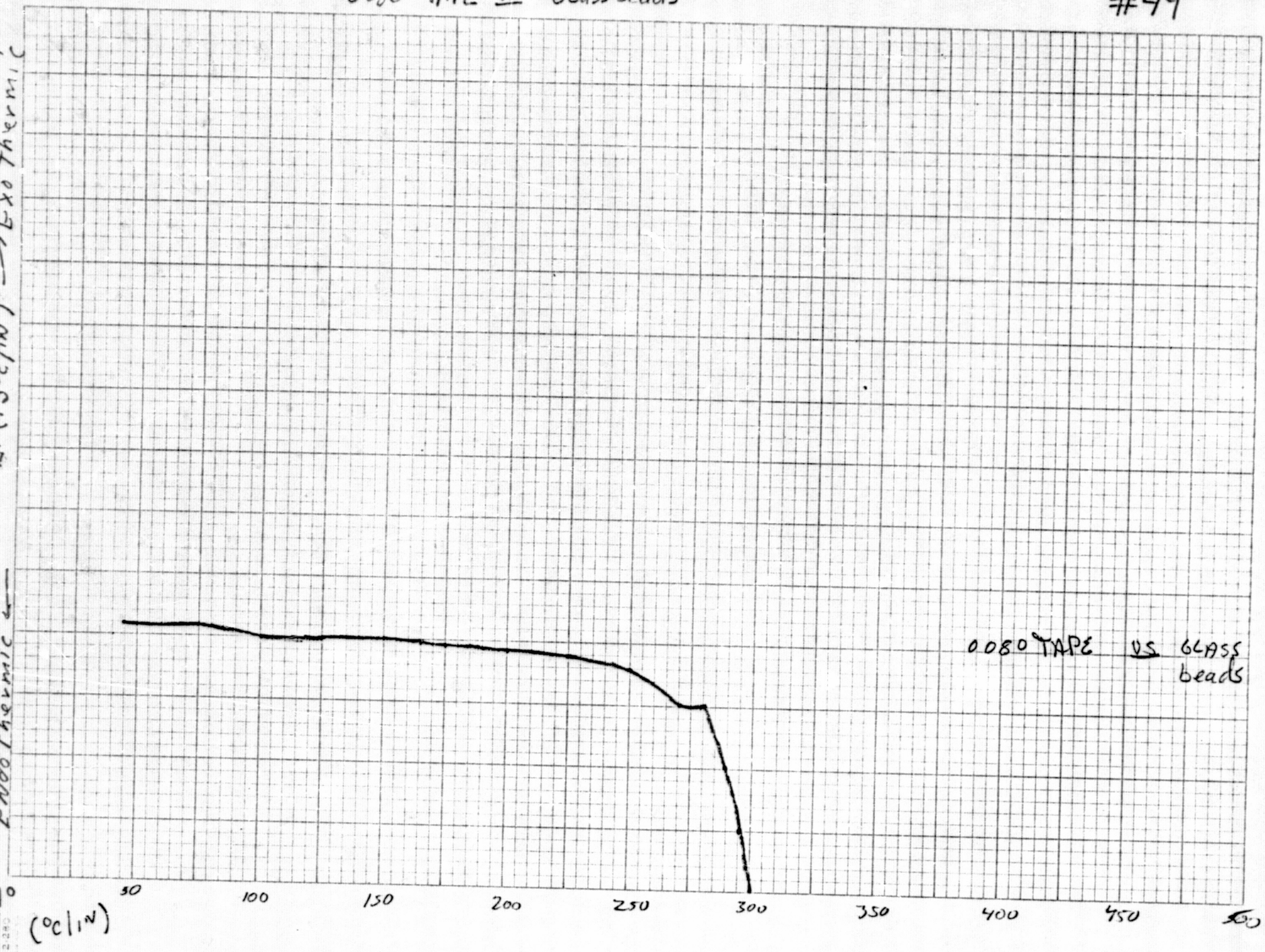


R.K.

10 Squares to the Inch

0080 TAPE vs Glassbeads

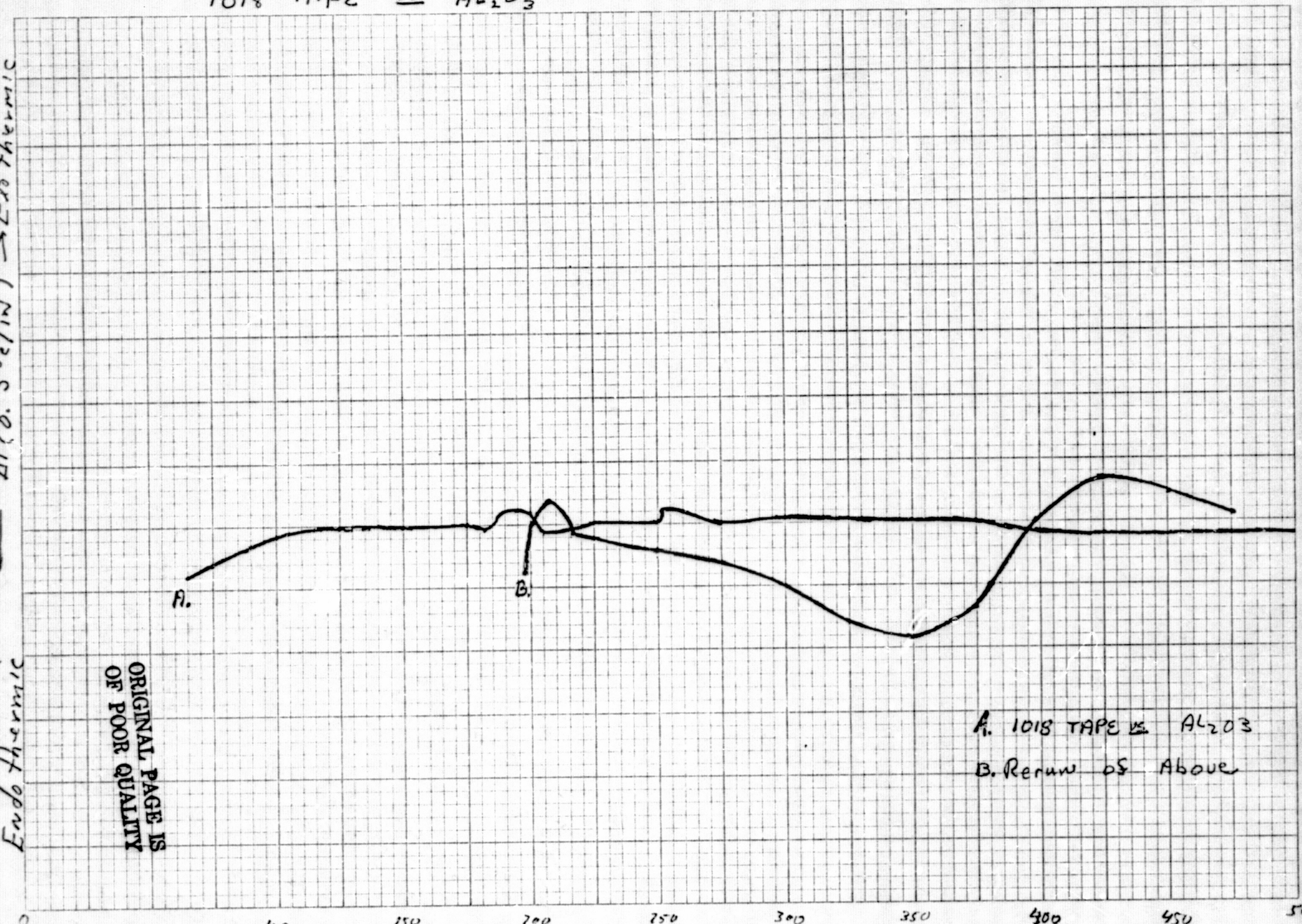
#44



KK

#5

1018 TAPE vs Al_2O_3



A. 1018 TAPE vs Al_2O_3

B. Rerun of Above

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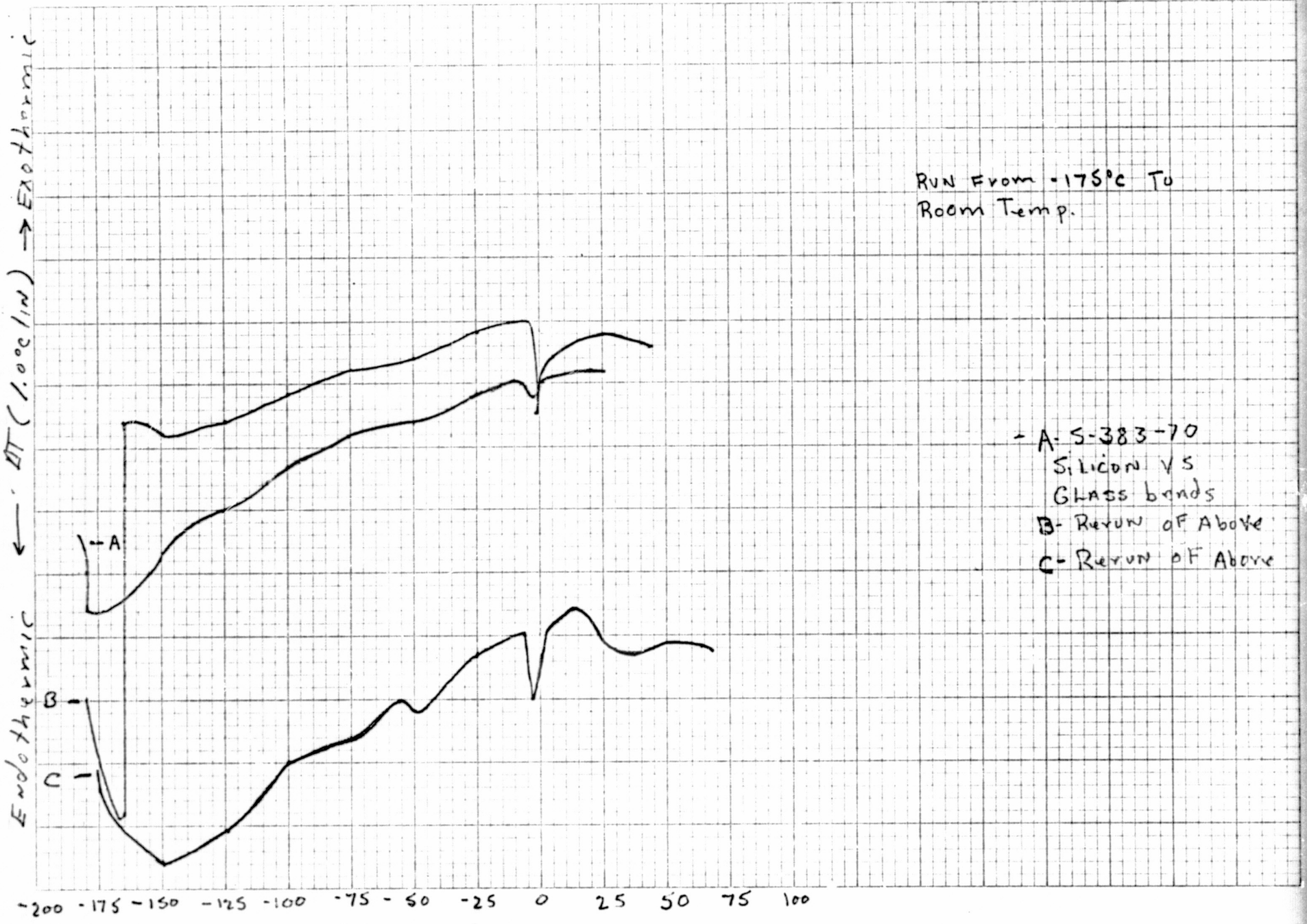
RK

S-383-70 SILICON VS GLASS BONDS #54



RUN FROM -175°C TO ROOM TEMP.

- A- S-383-70 SILICON VS GLASS BONDS
- B- RERUN OF ABOVE
- C- RERUN OF ABOVE

R.K

#60

Ethylene propylene AF-E-332

← ΔT (1.0°C/in) → Exothermic

Endothermic

-200 -150 -100 -50 0 50 100 150 200 250 300

A, B

A- Ethylene propylene AF-E-332 vs glass beads

B. Rerun of above

ORIGINAL PAGE IS OF POOR QUALITY

F61

← Endothermic ← ΔT (1.0°C/in) → Exo Thermic

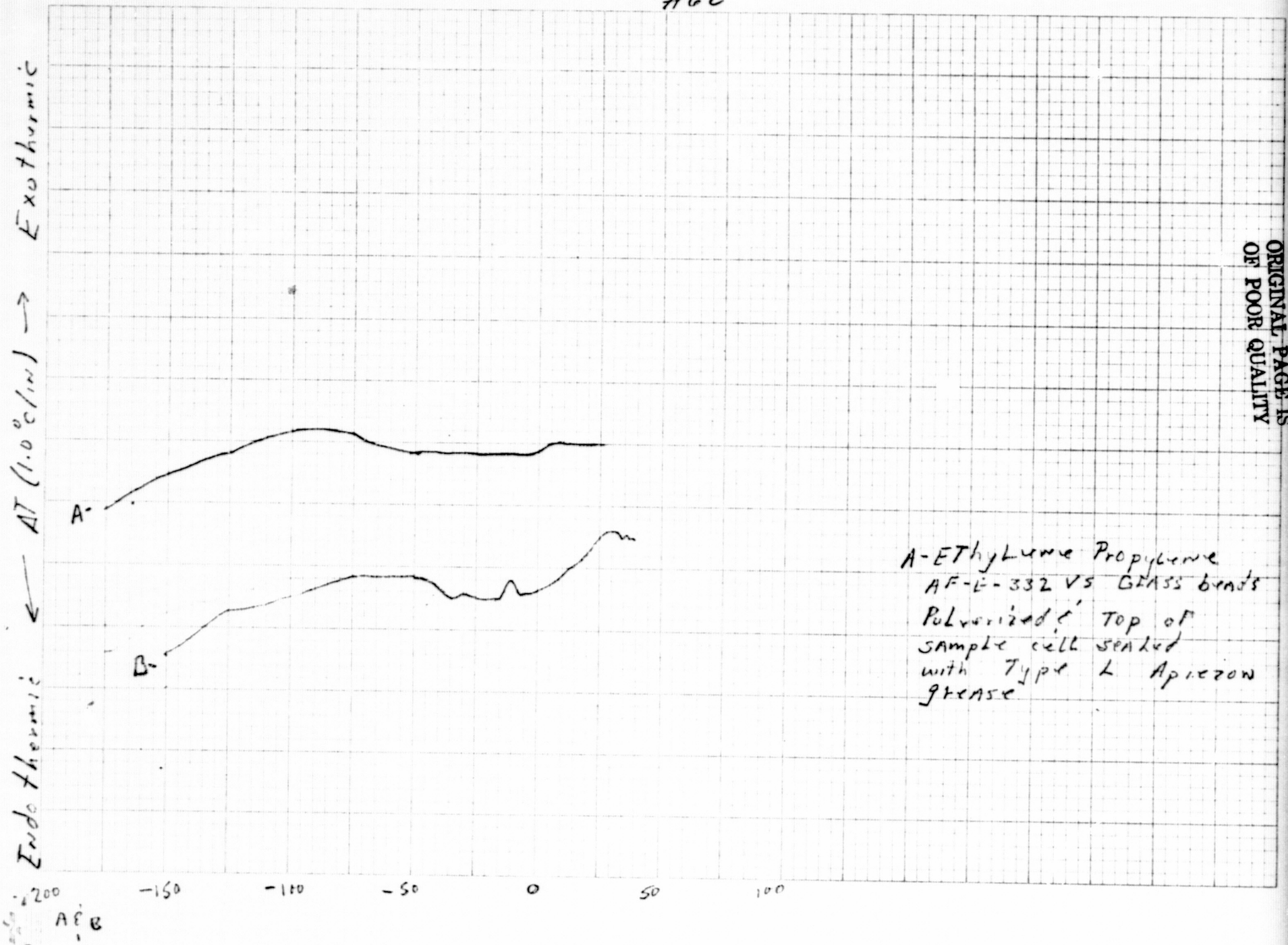
A- Ethylene Propylene
E-515-80 VS Glassbeads

B- Rerun of above

-200 -150 -100 -50 0 50 100 150

A, B

#162



A-Ethylene Propylene AF-E-332 vs GLASS beads Pulverized top of sample cell sealed with Type L Apiezon grease

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